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AIRBORNE DUST IN MINES
PUBLISHED REPORTS 1960-1987

G. Knight

Volume 1 MRL 88–114 (R)

MICROMEDIA

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AIRBORNE DUST IN MINES — PUBLISHED REPORTS: 1960-1987 CANMET/MINING RESEARCH LABORATORIES

Volume 1—SAMPLING AND ASSESSMENT

POUSSIÈRE AÉROPORTÉE DANS LES MINES— RAPPORTS PUBLIÉS: 1960-1987 CANMET/LABORATOIRES DE RECHERCHE MINIÈRE

ÉCHANTILLONAGE ET ÈVALUATION

G. Knight
Canadian Mine Technology Laboratory
Laboratoires canadiens de technologie Minière

OCTOBER 1987
OCTOBRE 1987

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394pp

CANMET MINING RESEARCH LABORATORIES
Divisional Report MRL 88–114 (R)

MICROMEDIA

FOREWORD

For many years an important aspect of the work of the Elliot Lake Laboratory has been studies of airborne dusts in underground mines. Several aspects have been addressed including: the development of instrumentation; in-situ studies; the characterization of airborne dusts; and, more recently, dust control and abatement.

Geoffrey Knight, one of the Division's senior scientists, has been associated with that work, often as project leader, for much of the period covered by this report. It was his initiative that resulted in the publications being brought together as a collection. It is our hope that this document will be useful both to the mining industry and to the universities in this important field.

John E. Udd
Director,
Mining Research Laboratories

AVANT-PROPOS

Depuis plusieurs années déjà, le Laboratoire d'Elliot Lake consacre une partie importante de ses activités à l'étude des poussières aéroportées dans les mines souterraines. À ce jour, les scientifiques se sont penchés sur plusieurs problèmes dont le développement de l'appareillage, les études in situ, la caractérisation des poussières aéroportées, et plus récemment, le contrôle et la réduction des concentrations de poussières.

Geoffrey Knight, un des scientifiques principaux de la Division, a participé aux recherches, maintes fois à titre de chef de projet, pendant la plus grande partie de la période couverte par ce rapport. Grâce à son esprit d'initiative, les publications ont pu être rassemblées en une collection. Nous espérons que le présent document constituera un outil précieux pour l'industrie minière et les universités qui s'intéressent à la recherche dans ce domaine.

John E. Udd
Directeur,
Laboratoires de recherche minière

Airborne Dust in mines — Published Reports:
CANMET/Mining Research Laboratories, 1960-1987

by

G. Knight*

ABSTRACT

This publication reproduces the reports published in journals, etc. by the Elliot Lake Laboratory, Dust Section, and by cooperating organisations on dust topics. The published documents, 35 in total, are given verbatim. They are divided into six main sections: 1, Dust Sampling and Assessment; 2, Dust Assessment; 3, Mine Studies; 4, Dust Characterization; 5, Dust Standards and Assessment Principles; and 6, General.

The three appendices list: 1, A list of the about 200 internal reports on dust topics that have been prepared; 2, A list of the major reports on control of diesel engine exhaust particulates; 3, A list of the research contracts carried out on dust topics on behalf of CANMET.

There is considerable overlap between the first and second sections. The first includes all papers on dust sampling and joint papers on dust sampling and assessment, the second includes those papers restricted to assessment. The split was introduced to balance the two volumes.

Keywords: Respirable dust; Dust sampling; Dust control; Dust measurement; Quartz assessment

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Contrôle de la poussière aéroportée dans les mines
Rapports publiés : CANMET/Laboratoires de recherche minière
1960-1987

par

G. Knight*

RÉSUMÉ

La présente publication comprend les rapports publiés sous forme d'articles de journaux, etc. par le Laboratoire d'Elliot Lake, Section de recherche sur la poussière, et par divers organismes qui collaborent à la rédaction d'articles sur le sujet. La documentation comprend, au total, 35 articles qui sont publiés textuellement et classés dans six catégories principales ; 1, Echantillonnage et évaluation de la poussière; 2, Évaluation des taux de poussière; 3, Études des conditions environnementales dans les mines; 4, Caractérisation de la poussière; 5, Normes relatives aux taux de poussière acceptables et principes d'évaluation; et 6, Généralités.

Les trois annexes comprennent : 1, Une liste d'environ 200 rapports de divisions portant sur le contrôle de la poussière; 2, Une liste des rapports principaux sur le contrôle des particules des gaz d'échappement diesel; 3, Une liste des contrats de recherche qui ont été réalisés pour le compte de CANMET dans le domaine du contrôle de la poussière.

La première et la deuxième section sont étroitement liées. La première section comprend tous les documents relatifs à l'échantillonnage de la poussière ainsi que des articles sur l'échantillonnage de la poussière et l'évaluation des taux de poussière acceptables et la deuxième inclut tous les documents qui ne portent que sur l'évaluation des taux de poussière. Les documents sont présentés de cette façon afin que les deux volumes se complètent.

Mots-clé : Poussière inhalable; Échantillonnage de la poussière; Contrôle des taux de poussière; Mesure des taux de poussière; Evaluation du quartz.

*Chercheur scientifique, Laboratoire canadien de technologie minière, CANMET, Énergie, Mines et Ressources Canada, Ottawa (Ontario).
Anciennement au service du Laboratoire d'Elliot Lake

TABLE of CONTENTS / TABLE des MATIÈRES

VOLUME 1–DUST SAMPLING AND ASSESSMENT / – CONTRÔLE DE LA POUSSIÈRE AÉROPORTÉE DANS LES MINES

FOREWORD i
AVANT-PROPOS iii
ABSTRACT v
RÉSUMÉ vii
TABLE of CONTENTS / TABLE des MATIÈRES ix
INTRODUCTION 1
ACKNOWLEDGEMENTS 2
INTRODUCTION en français 3
SECTION 1 DUST SAMPLING AND ASSESSMENT 1.1
  Chapter 1 Comparison of cyclone and horizontal elutriator size selectors 1.1
  Chapter 2 Comparison of dust sampling instruments 1.2
  Chapter 3 Full-shift assessment of respirable dust exposure 1.3
  Chapter 4 Development of a dust sampling system for hardrock mines based on gravimetric and quartz assessment 1.4
  Chapter 5 A combined personal sampler for dust and radon-daughter exposure in mines 1.5
  Chapter 6 Gravimetric dust sampling with quartz analysis and its use in metal and mineral mines 1.6
  Chapter 7 Mine dust sampling system–CAMPEDS 1.7
  Chapter 8 CAMPEDS – A dust sampling and assessment system for use in hard rock mines 1.8
  Chapter 9 Comparison of dust samplers: Statistical analysis techniques 1.9
  Chapter 10 Comparison of respirable dust samplers for use in hard rock mines 1.10

VOLUME 2–DUST ASSESSMENT, IN-MINE STUDIES, ETC. / – ÉVALUATION DE LA POUSSIÈRE, DES INSTALLATIONS MINIÈRES, ETC.

FOREWORD i
AVANT-PROPOS ii
ABSTRACT iii
RÉSUMÉ iv
TABLE of CONTENTS / TABLE des MATIÈRES v
INTRODUCTION 1

ACKNOWLEDGEMENTS 2
INTRODUCTION en français 3

SECTION 2 DUST ASSESSMENT (yellow separating pages) 2.11
Chapter 11 The electronic scanner - An instrument for the rapid counting of konimeter dust spots 2.11
Chapter 12 Determination of respirable quartz in mine air using personal samplers and X-ray diffraction 2.12
Chapter 13 The determination of quartz in dusts by infrared spectroscopy ... 2.13
Chapter 14 Laboratory calibration of a technique for determination of respirable quartz in mine air 2.14
Chapter 15 X-ray diffraction analysis of quartz in respirable dust: Brief description and procedure 2.15
Chapter 16 Overlap problems in counting fibers 2.16
Chapter 17 The use of an on-line computer to control quantitative XRD analysis 2.17
Chapter 18 Interlaboratory crosscheck of quartz analysis on mine airborne dust samples 2.18

SECTION 3 IN-MINE STUDIES (Red separating pages) 3.19
Chapter 19 Generation and control of mine airborne dust 3.19
Chapter 20 Dust control for scooptram operations 3.20
Chapter 21 How air quality can be monitored in an underground uranium mine 3.21
Chapter 22 Generation and control of airborne dust 3.22
Chapter 23 Computer predicted airborne respirable dust concentrations in mine air recirculation systems 3.23

SECTION 4 DUST CHARACTERIZATION (Green separating pages) 4.24
Chapter 24 A simple method for determining size distribution of airborne dust by its settling velocity 4.24
Chapter 25 Application of EDXRF analysis to the determination of metallic elements in airborne mineralogical dusts 4.25
Chapter 26 Size distribution of airborne dust in mines 4.26
Chapter 27 Size distribution of airborne dust in Labrador iron mines 4.27

SECTION 5 DUST STANDARDS AND ASSESSMENT PRINCIPLES

(Blue separating pages) 5.28
Chapter 28 Size selective sampling 5.28
Chapter 29 Routine dust measurements and standards 5.29
Chapter 30 Personal dust sampling in mines: Statistical analysis 5.30
Chapter 31 Comparison of respirable dust specifications with recent lung data 5.31
Chapter 32 Workplace respirable dust monitoring - Current and proposed canadian practice 5.32
Chapter 33 Rock fibre in nonasbestos mines - A suggested program for canadian mines 5.33
Chapter 34 Letters on alveolar dust deposition 5.34

SECTION 6 GENERAL (Orange seperating pages) 6.35
Chapter 35 Mines Branch dust laboratory 6.35
Chapter 36 Dust-sampling research and its application to mining 6.36
Chapter 37 Airborne contaminants and ventilation in mines 6.37

APPENDIX A Listing of CANMET Internal Reports; Titles only A.761
APPENDIX B Diesel Emission Control Studies B.789
APPENDIX C Contract Research undertaken for CANMET C.791

INTRODUCTION

The underground-environment dust studies have, over the past 27 years, been directed at three main topics: dust measurement instrumentation; in mine studies and dust characterization. The studies carried out under dust measurement instrumentation have had four major objectives. First, the comparison of the types of dust sampling instruments in wide use throughout the world in pure mineral dust clouds prepared in the laboratory. These studies were mainly carried out in the period 1965 to 1970 and culminated in the CANMET report R250 (1971). Second, the development of dust sampling techniques for routine use in mines. The main emphasis has been on a combined size-selective sampling and quartz analysis technique culminating in the CANMET Report 78-7 (1978). This technique was intended for routine use in mines and has been adopted,with modifications, in many Canadian mines. Third, continuous monitoring could be a useful technique for improving mine environment and some studies have been carried out. Fourth, Asbestos fibre in asbestos mines and mills as well as other mines is a potential health hazard and a limited effort has been directed to it. The potential health effects have been a major limiting factor in commercial use of asbestos and thus in Canadian production and exports.

The in-mine studies have been directed to three major objectives: Measurement of dust levels using the newly developed technique, gravimetric with quartz assessment; identification and quantification of the major dust sources in mines; and dust control techniques.

Dust characterization has been regarded as having long term objectives after developing and applying dust sampling techniques capable of determining the first order exposures of miners to dust namely total respirable dust and respirable quartz dust. The studies carried out have essentially been spare time, combined with other studies or directed at special problems.

The published papers are given verbatim in chronological order in each of six categories as follows:

1. Dust Sampling and Assessment
2. Dust Assessment
3. In-Mine Studies
4. Dust Characterization– composition and size distribution
5. Dust Standards and Assessment Principles
6. General

Dust instrumentation was split into two categories to balance the two volumes. There is overlap between the two and the first section includes the papers on dust sampling and on both sampling and assessment; the second is those restricted to assessment. Because the papers are reproduced as published the page numbering is erratic and each paper is treated as a chapter in a section. The chapters in each section are numbered consecutively with both section and chapter numbers. Each chapter has a coloured title page, each section uses a different colour. This is followed by a listing of the many internal reports by both MRL and other cooperating CANMET laboratories on underground- environment dust topics. This listing is an expansion of that given by M.G. Grenier and K.C. Butler in 'Index of underground dust reports: CANMET/Mining Research Laboratories,1960-1985' CANMET Special Publication SP86-4 (1986).

This report does not include all the dust related research carried out by the Canadian Explosives Atmospheres Laboratory of MRL in their Diesel engine exhaust control program or the coal mine dust studies carried out by the CANMET Coal Research Laboratory. A list of the major papers on heavy-duty diesel emission control is given in appendix B.

Appendix C lists the studies carried out by other organisations under contract to CANMET relevant to the dust topics included.

ACKNOWLEDGEMENTS

The author wishes to acknowledge the assistance of many colleagues and cooperating organisations in carrying out many of the studies reported in these papers. In particular the following organisations have made major contributions: Mines Accident Prevention Association of Ontario; McIntyre Research Foundation; the Labrador Institute of Northern Affairs, Memorial University; and the Quebec Asbestos Mining Association. The contributions of the staff from many mines has been very helpful.

INTRODUCTION

Les recherches réalisées au cours des vingt-sept dernières années sur le contrôle de la poussière dans les mines souterraines portaient sur trois sujets principaux : le développement d'un appareillage pour mesurer les concentrations de contaminants, l'environnement minier, et la caractérisation de la poussière. Les études portant sur des instruments de mesure des concentrations de poussière visaient quatre objectifs. D'une part, on a cherché à établir une comparaison entre les instruments d'échantillonnage de la poussière utilisés à l'échelle mondiale au moyen de nuages de poussière minérale préparée en laboratoire. La plupart des études ont été menées dans la période de 1965 à 1970 et ont abouti à la publication par CANMET du Rapport R250 (1971). D'autre part on a tenté de développer des techniques d'échantillonnage de la poussière pouvant être utilisées régulièrement dans les mines. Les recherches ont porté principalement sur le développement d'une technique d'échantillonnage d'après la taille des grains des particules combinée à la technique d'analyse du quartz. Un compte rendu des travaux de recherche a été publié dans le rapport 78-7 (1978) de CANMET. Cette technique est destinée à être utilisée régulièrement dans les mines et a été sélectionnée, moyennant quelques modifications, pour utilisation dans plusieurs mines canadiennes. En troisième lieu, la surveillance en continu qui pourrait s'avérer une technique utile pour améliorer l'environnement minier, à fait l'objet de certaines études. Enfin, les particules de fibres d'amiante dans les mines d'amiante et dans les installations de broyage de même que certaines installations minières constituent des sources potentielles de danger et sont à l'étude. Les effets néfastes qu'elles pourraient avoir sur la santé des travailleurs ont été le facteur principal sur lequel on s'est fondé pour limiter la production commerciale de l'amiante au Canada de même que l'exportation du produit.

Les études qui ont été réalisées dans les mines visaient trois objectifs : la mesure des concentrations de poussière au moyen de la nouvelle technique, l'analyse gravimétrique y compris l'évaluation du quartz; l'identification et la quantification des sources principales de poussière dans les mines; et les techniques de contrôle des concentrations de poussière.

Les recherches portant sur la caractérisation de la poussière comportent des objectifs à long terme. Cependant, on doit, au préalable développer et appliquer des techniques d'échantillonnage capables de déterminer chez les mineurs les premiers signes d'exposition aux contaminants, y compris l'exposition à la poussière entièrement inhalable et à la poussière de quartz. Les études ont presque toutes été réalisées à temps perdu, dans le cadre de recherches sur d'autres sujets ou sur des problèmes particuliers.

Les documents sont présentés dans l'ordre chronologique et classés dans l'une des six catégories qui suivent :

1. Échantillonnage et évaluation des concentrations de poussière
2. Évaluation des concentrations de poussière
3. Évaluation des conditions environnementales dans les installations minières
4. Caractérisation de la poussière - composition et distribution granulométrique
5. Normes relatives au contrôle de la poussière et principes d'évaluation
6. Généralités

Les articles sur les instruments de mesure des concentrations de poussière sont répartis en deux catégories de façon à ce que les deux volumes se complètent. La première et la deuxième section sont étroitement reliées, la première section comprenant des articles sur l'échantillonnage de la poussière de même que sur l'échantillonnage et l'évaluation tandis que la deuxième section porte uniquement sur l'évaluation. Les documents étant réimprimés sous leur forme originale, le numérotage des pages n'est pas ordonné et chaque document constitue un chapitre. Dans chacune des sections, les numéros des chapitres se suivent. La page titre de chacun des chapitres apparaît en couleur, et une couleur particulière différencie chacune des sections. Une liste des nombreux rapports de divisions préparés par LRM ou d'autres laboratoires qui collaborent à la rédaction d'articles sur le contrôle de la poussière dans le milieu minier souterrain est ensuite présentée. La liste complète celle qui a été compilée par M.G. Grenier et K.C. Butler lors de la puiblication du "Répertoire des rapports sur le contrôle de la poussière : CANMET/Laboratoires de recherche minière, 1960-1985" Publication spéciale de CANMET-SP86-4 (1986).

Le présent rapport ne comprend pas tous les articles sur le contrôle des concentrations de poussière publiés par le Laboratoire canadien de recherche sur les atmosphères explosives de LRM dans le cadre du programme sur le contrôle des émissions diesels ou les rapports des études sur le contrôle de la poussière dans les mines réalisées par le Laboratoire de recherche sur le charbon de CANMET. Une liste des documents les plus importants qui ont été publiés sur le contrôle des émissions diesels est présentée à l'annexe B.

L'annexe C comprend le répertoire des études effectuées en sous-traitance pour le compte de CANMET par d'autres organismes et qui ont rapport aux sujets traités.

REMERCIEMENTS

L'auteur de ce rapport désire remercier ses collègues et les organismes qui ont participé aux recherches dont il est question dans ces documents. La collaboration des organismes suivants a été des plus précieuses : Mines Accident Prevention Association of Ontario; McIntyre Research Foundation; the Labrador Institute of Northern Affairs, Memorial University; et l'Association des mines d'amiante du Québec. La participation du personnel de plusieurs installations minières a également été très appréciée.

LISTING OF PAPERS: Section 1.
Dust Sampling and Assessment

1. 1

Section 1—Chapter 1

Comparison of Cyclone and Horizontal Elutriator Size Selectors

by

G. Knight and K. Lichti

The American Industrial Hygiene Association Journal
31(4) pp437-41 (1970)

COMPARAISON DES CLASSIFICATEURS À CYCLONE ET À ÉLUTRIOMÈTRE HORIZONTAL

G. KNIGHT et K. LICHTI

Ministère de l'Énergie, des Mines et des Ressources
Direction des mines
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On a comparé des échantillonneurs de poussière équipés de deux types de classificateur, le cyclone et l'élutriomètre horizontal, dans une large gamme de nuages de poussière minérale. Les résultats montrent que le classificateur à cyclone (hydrocyclone de 10 mm) fonctionnant avec un débit de 1,4 litres/minute donne une concentration massique de poussière "inhalable" approximativement égale à celle obtenue avec l'élutriomètre horizontal construit selon les recommandations du Medical Research Council (Royaume-Uni). Cette concentration est aussi très proche de celle obtenue en utilisant les recommandations de l'Atomic Energy Commission (États-Unis).

Comparison of Cyclone and Horizontal Elutriator Size Selectors

G. KNIGHT and K. LICHTI

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Dust samplers fitted with two types of size selector, the cyclone and the horizontal elutriator, were compared in a wide range of mineral dust clouds. Results indicate that the cyclone size selector (10-mm hydroclone) when operated at 1.4 liters/min gave a "respirable" dust concentration by mass approximately equal to that obtained by using the horizontal elutriator built to the Medical Research Council (United Kingdom) recommendations. This cyclone at this flow rate also gives a "respirable" mass concentration corresponding closely to that obtained by using the Atomic Energy Commission (United States) recommendations.

Introduction

THERE IS CONSIDERABLE interest in the use of gravimetric samplers for assessing the pneumoconiosis hazard of airborne dust clouds. For this purpose it is essential to use a size selector to reject the coarse dust particles that do not penetrate to the depths of the lung and that could otherwise make a large contribution to the mass of dust collected. A number of experimental studies have been made of the size of dust particles deposited in the lungs. These have been reviewed by Davies,[1] who puts forward the curve of lung deposition shown in Figure 1, and also by the U. S. Atomic Energy Commission Meeting on sampling respirable dust, reported by Lippmann and Harris.[2] It is stated that lung deposition is affected by many factors, and for the design of a dust-sampling instrument these need to be averaged.

Two recommendations have been made for the specification of size selectors for sampling respirable dust, both shown in Figure 1. The first, by the United Kingdom Medical Research Council (MRC),[3] was chosen to suit the possibilities of the horizontal elutriator, and to enable the exploitation of its advantages. The horizontal elutriator offers a well-defined and reproducible performance, adaptability to instruments operating at widely different flow rates, and insensitivity to small dimensional variations in construction. The theory of the horizontal elutriator was given by Walton[4] and confirmed experimentally by Hamilton.[5]

The second size-selector specification, recommended by the U. S. Atomic Energy

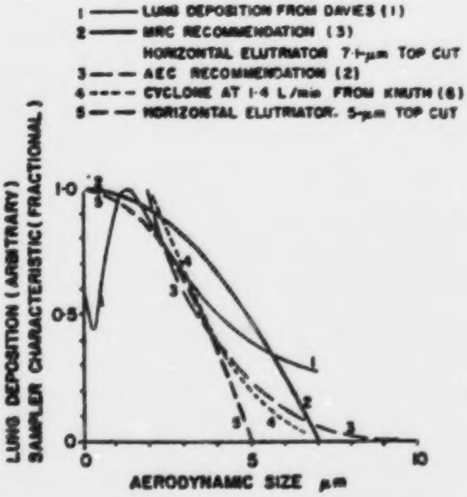

FIGURE 1. Lung deposition, size-selective sampling recommendations, and size-selector characteristics. 1——lung deposition, from Davies.[1] 2——MRC recommendation,[3] standard elutriator, 7.1-μm top cut. 3— —AEC recommendation.[2] 4- - - cyclone at 1.4 liters/min, from Knuth.[6] 5— —fine elutriator, 5-μm top cut.

Commission (AEC) Meeting on sampling respirable dust,[3] though possibly nearer to the average lung-deposition curve, has not yet been matched by a size selector. A cyclone can have a size-selection characteristic close to the AEC recommendation as shown in Figure 1, after Knuth,[4] but unfortunately the performance of a cyclone cannot be defined theoretically. Accurate calibration is difficult, as is shown by the widely different airflow rates recommended in two different calibrations of the 10-mm nylon hydroclone (Dorr-Oliver cyclone, unmodified, supplied by Union Industrial Equipment Co.) when used as a size selector.[3,4]

Cyclones are, however, of considerable interest as size selectors, because they are, for a given flow rate, considerably smaller than horizontal elutriators and because they do not have a fixed orientation. These characteristics are particularly useful in personal dust samplers.

The object of the work reported is threefold:

1. To calibrate the 10-mm nylon cyclone against the MRC "respirable" dust specification by comparison with the horizontal elutriator.
2. To determine the reproducibility of the cyclone estimate of the MRC "respirable" dust concentration in the range of size distribution and material type likely to occur in mine dust clouds.
3. To compare the MRC and AEC specifications for respirable dust by comparing the calibration in this report with that given by Knuth[4] of the AEC Health and Safety Laboratory.

Because of the belief by some people that the Medical Research Council size-selection curve is too coarse, comparisons were also made with a modified (fine) horizontal elutriator with a 5-μm top cut instead of a 7.1-μm top cut (MRC standard elutriator). The top cut is the maximum size of particle that can penetrate the elutriator.

The term "selected dust concentration" is used in this report to indicate the dust concentration determined by collection on the filter of a dust sampler fitted with a size selector. The term "aerodynamic size" of the particle is defined as the diameter of a unit-density sphere with the same settling velocity in air as the particle in question.

Experimental Techniques

Four 10-mm nylon cyclones were operated at airflows of 1.3, 1.65, 1.95, and 2.64 liters/min controlled by critical orifices at a set inlet pressure. These were compared with three MRC standard horizontal elutriators operated at 50-, 2.83-, and 2.5-liters/min airflows and with one "fine" horizontal elutriator (5.0-μm top cut) operated at 25 liters/min.

The comparisons were carried out in a dust test chamber on thirty dust clouds of thirteen types. These were chosen from the types of laboratory dust clouds used at Elliot Lake, to cover a wide range of size distributions, densities, and shapes.

Most of the comparisons were carried out for a sampling period of 40 minutes, a few at higher concentrations were run for less time, and some at lower concentrations were extended to 80 minutes. The mass of dust collected on the cyclones was between 0.3 and 1.5 mg and was weighed to 0.02 mg.

Results

Measurements were made in thirty dust clouds; the "selected" dust concentrations obtained are shown in Table I. The total dust concentrations, measured by an open filter, ranged from 1.2 to 2.5 times the selected mass determined by the standard horizontal elutriator. To compare the cyclones with the horizontal elutriators, a statistical analysis of the ratios of selected dust concentrations was carried out. The resultant means and standard deviations are shown in Table II and plotted against the airflow through the cyclone in Figure 2. It can be seen that the plot of the mean ratio between the cyclones and the MRC standard elutriator crosses the unit ratio at a flow rate of 1.4 liters/min.

Thus, samplers fitted with either an MRC standard elutriator or a 10-mm cyclone operated at 1.4 liters/min give the same estimate of selected dust concentration with a standard deviation of about 10%. Similarly, samplers fitted with a "fine" elutriator or a cyclone operated at 2.05 liters/min give the

TABLE I

Dust Measurements Using Elutriator and Cyclone Size Selectors

	"Selected" Dust Concentration (mg/m³)							
	Horizontal Elutriators				Cyclones			
	"Fine"	MRC Standard						
	Airflow (liters/min)							
Dust Cloud.	25	50	2.83	2.5	2.64	1.95	1.65	1.3
Coal								
Jet-milled	15.3	16.8	17.8	17.9	10.9	17.1	—	18.0
	19.5	21.4	20.6	22.6	16.6	25.0	—	25.6
Cyclone	14.1	20.3	18.5	20.2	10.1	13.6	—	22.9
	15.3	21.1	21.2	24.0	13.2	16.4	—	24.9
Silica								
Jet-milled	14.35	16.1	18.8	17.5	12.2	15.4	15.3	18.1
	10.6	11.9	12.1	12.2	9.0	11.2	10.2	13.7
	9.90	10.75	13.0	11.7	10.3	13.4	13.5	14.2
	10.9	12.1	13.4	13.4	8.3	11.5	13.6	15.0
Pulverized	13.0	19.0	17.1	18.4	10.7	14.6	16.7	19.6
	12.9	18.5	17.7	18.2	9.95	13.8	15.8	20.2
Cyclone	11.1	18.9	16.3	17.0	7.8	12.3	13.6	16.2
	10.6	15.5	15.6	14.6	7.8	10.5	13.2	15.8
	5.3	6.7	7.0	—	4.9	6.4	6.8	7.1
	8.8	11.8	11.5	12.6	7.1	8.6	10.2	11.9
No cyclone	10.9	16.3	16.0	17.1	9.1	11.9	13.6	18.8
	10.9	16.4	15.8	16.1	7.9	11.8	12.1	16.9
Pyrite								
Jet-milled	43.8	32.7	46.5	33.4	—	45.7	47.4	53.1
	18.7	21.0	21.7	21.6	16.0	18.8	20.0	24.4
Cyclone	3.3	3.3	3.5	3.6	2.75	3.9	4.4	6.35
	3.8	6.0	5.6	3.25	3.1	4.4	4.6	3.6
	3.5	5.7	5.16	3.8	2.4	3.3	3.6	4.7
Mica								
Jet-milled	16.1	19.8	19.0	20.3	15.2	16.2	19.6	20.3
	17.05	20.95	21.95	23.3	—	—	22.2	23.9
Glass fiber								
Jet milled	3.4	3.9	3.7	3.7	2.8	3.0	3.4	4.0
Pulverized	1.6	2.25	1.85	1.9	1.45	1.7	2.3	2.3
	.97	1.21	.91	1.1	.48	.8	.77	1.1
Asbestos								
Jet-milled	12.6	13.6	13.75	12.0	11.8	12.05	11.8	11.2
	12.5	13.3	12.7	13.0	9.5	11.5	12.2	12.7
Pulverized	4.2	5.4	4.9	5.4	2.8	4.2	4.2	4.8
	5.2	6.6	6.0	6.6	4.3	5.0	5.0	6.2

same estimate of the selected dust concentration with a standard deviation of about 12%.

There is a suggestion of a size-distribution effect on some of the comparisons. In Figure 3, for example, the selected dust concentrations from the cyclones relative to those from the MRC standard elutriator have been plotted against the mass median size (based on settling velocity). The significance of the regression line for the nonfibrous dust clouds (shown in the figure) is 0.1. Thus a size effect is suggested, but not established. The regression line corresponds to about a 10% change in the ratio of masses collected over the range of dust clouds likely to be found in mines.

Discussion

The results obtained show that a cyclone can be used to give a reasonable estimate of the "respirable" mass concentration to the MRC specification. This estimate has a standard deviation of 10%, which may be due to experimental error and in part to systematic

TABLE II

Comparison of Cyclone and Horizontal-Elutriator Size Selectors

Cyclone Flow Rate (liters/min)	Ratio of "Selected" Dust Concentrations	
	Mean	Standard Deviation
	Sampler Fitted with Cyclone / Sampler Fitted with MRC Elutriator	
1.3	1.06	0.116
1.65	0.90	0.134
1.95	0.83	0.128
2.64	0.64	0.14
	Sampler Fitted with Cyclone / Sampler Fitted with Fine Elutriator	
1.3	1.34	0.229
1.65	1.13	0.163
1.95	1.05	0.110
2.64	0.795	0.112

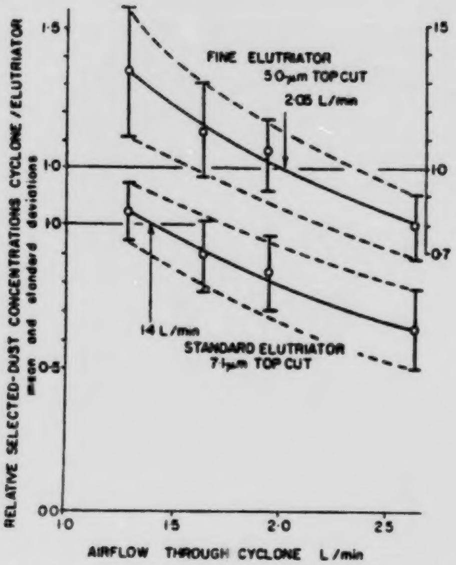

Figure 2. Comparison of cyclone and horizontal-elutriator size selectors.

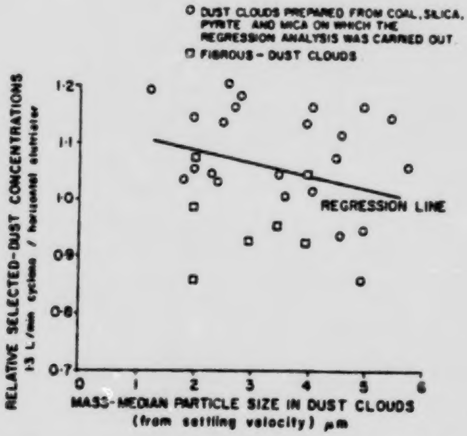

Figure 3. Effect of size distribution on size-selector comparison.

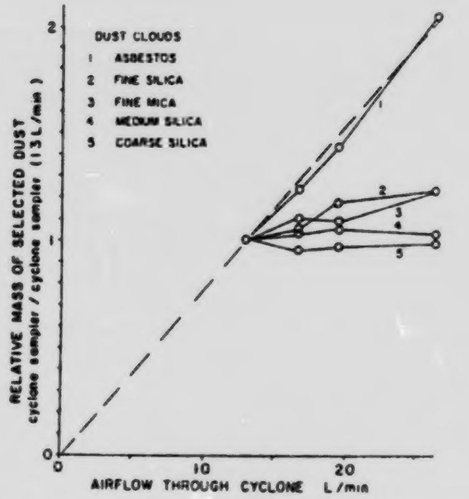

Figure 4. Effect of airflow on mass of dust collected by cyclones.

effects of size distribution. An estimate of the experimental errors may be obtained from the comparison of the estimates by the three MRC standard elutriators. This comparison gave a standard deviation of 7%, leaving, because statistical variance is additive, an estimate of 7% for the standard deviation of the systematic effects in the comparison of cyclones and horizontal elutriators. This variation is not too great to prevent an adequate determination of the "respirable" dust concentration in mines by a cyclone, when the other sources of variation, especially that with time, are considered.

Though this conclusion is based on a study of only one type of cyclone, it is believed that any cyclone will give an adequate estimate with a similar variance if its flow rate is adjusted to give the same respirable concentration as that given by a horizontal eutriator.

The closeness of the MRC and the AEC specifications for respirable dust is shown by the same flow rate, 1.4 liters/min, being found when calibrating the cyclone to the MRC specification (this report) or to the AEC specification.[6] The author concludes that in mines the two specifications are interchangeable and that the best field method of checking a cyclone size selector is to compare the "selected" dust concentration collected with that obtained using a horizontal elutriator. Large differences between the "respirable" dust concentration based on the two specifications could arise only in dust clouds with a very sharp size distribution. These, however, have not been reported in mine and industrial atmospheres and, in the author's opinion, are not likely to be found there.

One advantage of cyclone size selectors, as contrasted to horizontal elutriators, is that errors in airflow are compensated to some extent by changes in efficiency. Figure 4 shows the selected mass of dust collected by the four cyclone samplers for four dust clouds covering the range of size distributions. It can be seen that in dust cloud No. 1 the mass collected on the filter is proportional to the flow rate, but that in the others it is approximately independent of flow rate. Dust cloud No. 1 is unusual in that it consists of three components—fine particles, fine fibers, and coarse-matted fibrous particles—with very little dust in the 3- to 10-μm size range.

Conclusions

It is concluded that:

1. Dust samplers fitted with MRC standard horizontal elutriator or 10-mm nylon cyclone size selectors give the same mean estimate of the "respirable" dust concentration by mass when the cyclone is operated at 1.4 liters/min.

2. The estimate of "respirable" dust concentration by mass determined by a dust sampler fitted with a cyclone size selector is essentially the same when calibrated against either the United Kingdom MRC or the United States AEC specifications.

3. The size distribution may have a systematic effect on the comparison of the two size selectors. This amounts to less than a 10% change in the mass estimated in the range of size distributions likely to be found in mine dust clouds.

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Received August 27, 1969

Section 1—chapter 2

Comparison of Dust Sampling Instruments

by

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Mines Branch Research Report R250 (1971)
Energy Mines and Resources Canada, Mines Branch
Ottawa, Ontario

COMPARISON OF DUST SAMPLING INSTRUMENTS

by

ENVIRONMENTAL CONTROL GROUP*

SUMMARY

This report describes many comparisons between dust sampling instruments. These narrow down to the very wide range in ratio of dust concentrations indicated by any two types of dust sampling instruments in different dust clouds and, therefore, the need to consider physiological factors in applying dust sampling instruments to assess health hazards. Changes in design that are apparently minor can have a large effect on the respirable dust concentration indicated by a sampler that has an aerodynamic size selector.

Dust Sampling: Thermal Precipitator: Midget Impinger: Tyndalloscope: Gravimetric Sampling: Size Selection: Respirable Dust.

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Direction des mines

Rapport de recherches R 250

LA COMPARAISON DES INSTRUMENTS POUR L'ÉCHANTILLONNAGE DES POUSSIÈRES

par

Le Groupe de Contrôle de l'Environnement*

RÉSUMÉ

Dans ce rapport, les auteurs décrivent plusieurs comparaisons entre les instruments pour l'échantillonnage des poussières. Celles-ci se limitent à un champ étendu dans le rapport des concentrations de poussière indiquées par deux de n'importe quels genres d'instruments pour l'échantillonnage des poussières dans les différents nuages de poussière et, de là, le besoin de considérer les facteurs physiologiques dans l'application des instruments pour l'échantillonnage des poussières en vue d'évaluer les dangers pour la santé. Les changements de dessin qui sont apparemment mineurs peuvent avoir un grand effet sur les concentrations de poussière respirable indiquées par un échantillonnage qui a un sélecteur de taille aérodynamique.

L'Échantillonnage des poussières: Précipitateur thermique: "Midget Impinger": Tyndalloscope: L'Échantillonnage gravimétrique: Sélection de taille: Poussière respirable.

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CONTENTS

Page

Chapter 1 - Airborne Dust Sampling
Introduction -- 1-1
Phsiological Background 1-1
Deposition of Dust in the Lungs 1-1
Clearance of Dust from the Alveolar Region in the Lungs 1-2
Biological Effect of Dust in the Lungs 1-4
Scope of this Report 1-4
References 1-4

Chapter 2 - Experimental Techniques
Dust Chamber 2-1
Dust Sampling Instruments 2-1
Instruments Assessing Dust by Number 2-2
Instruments Assessing Dust by Light Scatter 2-6
Instruments Assessing Dust by Mass 2-7
Respirable Dust Size Selection 2-7
Airflow Control and Calibration 2-8
The Dust Clouds 2-9
Techniques for Analysis of Results 2-10
References 2-12

Chapter 3 - Comparisons between Instruments Assessing Dust by Number
Accuracy of Dust Estimates 3-1
The Instrument Comparison Indices 3-4
The Effect of Concentration 3-4
The Non-Linearity of Comparisons with the Konimeter -- 3-4
The Effect of Particle Size Distribution 3-6
The Effect of Density 3-8
The Effect of Particle Shape 3-8
The Effect of Aggregation 3-8
Comparison of Microscope Techniques 3-10
Comparison of the Collection Efficiencies 3-11
Comparison of Counting Size Ranges on Thermal Precipitator Samples 3-16
The Effect of Heat Treatment and Acid Wash on Konimeter Dust Estimates 3-16
References 3-16

Chapter 4 - Comparison between Instruments Assessing Dust by Number, by Light Scatter, and by Mass

- Accuracy of Measurement by Each Instrument Type ---------- 4-4
- The Comparison Indices ---------- 4-4
- The Effect of Concentration ---------- 4-4
- The Effect of Particle Size Distribution ---------- 4-5
- The Effect of Density ---------- 4-5
- The Effect of Particle Shape ---------- 4-5
- The Effect of Aggregation ---------- 4-12

Chapter 5 - Comparisons of Respirable Dust Size Selectors

- The Effect of Design Detail on the Performance of the Horizontal Elutriator Size Selector ---------- 5-1
- Variations in Performance of Cyclone Size Selectors ---------- 5-3
- Comparisons of Cyclone and Horizontal Elutriator Size Selectors ---------- 5-5
- Advantages ---------- 5-8
- Disadvantages ---------- 5-10
- References ---------- 5-10

Appendix A - Dust Cloud Preparation and Properties

- Introduction ---------- A-1
- Dust Sources ---------- A-1
- Dust Preparation ---------- A-2
- Dust Feeding ---------- A-3
  - USBM Type Feeder ---------- A-3
  - Wright Feeder ---------- A-5
  - SMRE Type Feeder ---------- A-6
- Dust Dispersion ---------- A-8
  - Jet Mill ---------- A-8
  - Pulverizer ---------- A-8
  - Compressed Air Ejector ---------- A-9
  - Atomization of Dust Suspension in Liquid ---------- A-9
- Top Size Control of Dust ---------- A-10
- Chamber Airflow ---------- A-10
  - Control Setting for Standard Dust Clouds ---------- A-12
- Some Properties of the Dust Clouds ---------- A-12
  - Variations of Concentration within the Chamber ---------- A-12
  - Variations of Dust Concentration with Time ---------- A-12
- Particle Shape ---------- A-18
- Size Distribution by Volume: Coulter Counter ---------- A-20
- Size Distribution: Optical Microscope ---------- A-20
- Size Classification by Falling Speed ---------- A-29

Surface Area -- A-30
References -- A-33

Appendix B - Statistical Analysis Technique
Introduction -- B-1
Experimental Design for the Comparison of Instruments ----- B-1
Method of Statistical Analysis -------------------------- B-2
The Reference Instrument ----------------------- B-4
Independent Variables for Types of Dust Clouds ------- B-4
Independent Variables for Instruments ------------- B-7
The Intercept ----------------------------------- B-7
The Computer Program -------------------------------- B-8
Presentation of Data and Controls --------------------- B-9
Presentation of Results ------------------------------- B-10
Discussion -- B-14
The Errors of the Independent Variables ------------ B-14
Conclusions --- B-18
Acknowledgements ------------------------------------ B-18
References -- B-19

Appendix C - Detailed Measurements of Dust in the Comparisons of Instruments -- C-1

Appendix D - Detailed Measurements of Dust Concentration Using Gravimetric Dust Samplers Fitted with Size Selectors ----- D-1

LIST OF FIGURES

1-1 Dust deposition inthe lungs, showing range of variation with the breathing rate (after Hatch and Gross) --- 1-3

2-1 Position of dust samplers in chamber (isometric projection) -- 2-3

3-1 Comparison between instruments assessing dust by number with best fit lines by statistical regression ------ 3-2

3-2 The effect of dust concentration on the relative concentrations estimated by the konimeter ------------- 3-7

3-3 Effect of particle size and density on comparison indices of dust sampling instruments ------------------------- 3-9

3-4 Collection efficiencies of impingers and konimeters compared to that of thermal precipitators -------------- 3-14

4-1 Mass number comparison --------------------------- 4-2

4-2 Effect of size classification, density, and shape on respirable mass/mean number comparison index -------- 4-9

4-3 Effect of size classification, density and shape on respirable mass/light scatter comparison index ---------- 4-10

4-4 Effect of size classification, density and shape on mean number/light scatter comparison index ------------ 4-11

5-1 Schematic diagrams and relative performance of respirable-dust samplers -------------------------- 5-2

5-2 Effect of airflow on respirable dust concentration estimated by samplers with cyclone size selectors ------- 5-6

5-3 Effect of dust size distribution on comparison between cyclone and horizontal-elutriator size selectors ---------- 5-7

5-4 Effect of airflow and dust size on the respirable-dust concentration estimated by samplers using cyclone ------ size selectors 5-9

A-1 Dust feed apparatus ---------------------------------- A-4

A-2 The SMRE type feeder ------------------------------- A-7

A-3 Airflow through chamber ---------------------------- A-11

A-4 Variation in dust concentration with time: 40 to 100-mesh coal fed by SMRE and dispersed by jet mill ------------- A-14

A-5 Variation in dust concentration with time: pulverized silica fed by SMRE and dispersed by air ejector ---------- A-15

A-6 Variation in dust concentration with time: asbestos fed by SMRE and dispersed by jet mill ----------------- A-16

A-7 Variation in dust concentration with time: pulverized silica fed by USBM and dispersed by air ejector --------- A-17

A-8 Size distribution of the coal dust clouds - Coulter Counter --- A-21

A-9 Size distribution of the silica dust clouds - Coulter Counter -- A-22

A-10 Size distribution of pyrite dust clouds - Coulter Counter ----- A-23

A-11 Size distribution of the mica dust clouds - Coulter Counter -- A-24

A-12 Size distributions of asbestos dust clouds - Coulter Counter - 140- and 50-μm-diameter orifices ------------- A-25

A-13 Size distributions of the glass fibre dust clouds - Coulter Counter - 140- and 50-μm-diameter orifices -------- A-26

B-1 The dependence of the errors on the dust concentration ------ B-3

B-2 Distribution of residuals from two regression analyses ----- B-15

LIST OF TABLES

3-1 Accuracy of Dust Estimates ------------------------- 3-3

3-2 Instrument Comparison Indices: Number/Number -------- 3-5

3-3 Distribution of Particles in Aggregated Dust Clouds ------- 3-10

3-4 Comparison of Microscope Techniques on Thermal Precipitator Slides ------------------------------- 3-12

3-5 Comparison of Thermal Precipitators with the Impingers and Konimeters Using the Impinger and Konimeter Microscope Techniques Respectively ----------------- 3-15

3-6 Comparison of Counting Size Range on Thermal Precipitator Slides ------------------------------ 3-17

4-1 Accuracy of the Dust Estimates by Each Instrument ------ 4-3

4-2 Instrument Comparison Indices between Mass Number and Light Scatter ------------------------------- 4-6

4-3 Instrument Comparison Indices: Mass/Number 4-7

4-4 Instrument Comparison Indices: Number/Light Scatter 4-8

5-1 Cyclone Size Selectors: Variations in Dust Collection 5-4

A-1 Pulley Size and Hoisting Speed A-5

A-2 Feed Rate of SMRE Type Dust Feeder A-6

A-3 Conditions for Producing Standard Dust Clouds A-13

A-4 Variation in Mean Dust Concentration between Runs (Hexhlett) A-19

A-5 Coulter Counter Log Normal Size Distributions Constants for Best Fit A-27

A-6 Size Classification by Optical Microscope A-28

A-7 The Size Distribution in the Fibrous Dust Clouds A-29

A-8 Experimental Determinations of Particle Size Distributions Using Size Selective (Horizontal Elutriator) Samplers A-31

A-9 The Specific Surfaces of Cloud Dusts A-32

Dust Concentration Measurements

C-1 Material: Coal: Dispersion Method: Jet Mill C-3
C-2 " Pulverizer C-4
C-3 " Ejector-Cyclone C-5
C-4 " Ejector-No Cyclone C-6
C-5 Silica: Jet Mill C-7
C-6 " Pulverizer C-8
C-7 " Ejector-Cyclone C-9
C-8 " Ejector-No Cyclone C-10
C-9 Pyrite: Jet Mill C-11
C-10 " Pulverizer C-12
C-11 " Ejector-Cyclone C-13
C-12 " Ejector-No Cyclone C-14
C-13 Mica: Jet Mill C-15
C-14 " Pulverizer C-16
C-15 " Ejector-Cyclone C-17
C-16 " Ejector-No Cyclone C-18
C-17 Asbestos: Jet Mill C-19

C-18 Asbestos: Pulverizer ---- ---- C-20
C-19 Glass Fibre: Jet Mill ---------- C-21
C-20 " Pulverizer ----- --- C-22
C-21 Aggregated Coal Dust Cloud: Atomizer (Full-Strength Suspension) C-23
C-22 Aggregated Coal Dust Cloud: Atomizer (Dilute) --- C-25
C-23 Extension to Low Concentrations; Material: Coal; Dispersion Method: Jet Mill ---------------------------- C-27

C-24 Dust Concentration Measurements: Extension to Low Concentrations; Material: Coal; Dispersion Method: Ejector-No Cyclone ---------------------------- C-28

C-25 Dust Concentration Measurements: Extension to Low Concentrations; Material: Silica; Dispersion Method: Jet Mill ---------------------------- C-29

C-26 Comparison of Dust Sampling Instruments and Microscope Techniques ---------------------------- C-30

D-1 Dust Concentration Measurements Using Elutriator and Cyclone Size Selectors ---------------------------- D-3

Dust Concentration Measurements

D-2 Material: Coal: Dispersion Method: Jet Mill---------- -- D-4
D-3 " Air Ejector-No Cyclone D-5
D-4 " Air Ejector - No Cyclone and Jet Mill - D-7
D-5 " Jet Mill ----------- D-8
D-6 " Air Ejector - No Cyclone ----------- D-9
D-7 " Air Ejector - No Cyclone ----------- D-11
D-8 " Jet Mill ----------- D-12
D-9 " (Low Concentration) - D-13
D-10 " Jet Mill D-14
D-11 " Air Ejector-No Cyclone D-15

CHAPTER 1
AIRBORNE DUST SAMPLING

INTRODUCTION

Airborne dust sampling is done in mines because it is a hazard to health. Dust sampling in mines is normally directed to one or more of the following objectives:

- assessment of the health hazard to which miners are exposed;
- control of dust sources;
- characterization of the dust cloud and relating to clinical observations.

PHYSIOLOGICAL BACKGROUND

Because the main hazard is pneumoconiosis in its many forms such as silicosis and anthracosis, dust sampling should be directed toward estimating the potential pneumoconiosis hazard of airborne dust. To do this, it is essential to consider the physiological processes involved in pneumoconiosis and to measure the appropriate parameters of the dust. The dust cloud is related to the onset of pneumoconiosis by:

the deposition of dust in the lungs,
the clearance of dust from the lungs, and
the biologic activity of dust in the lungs.

Deposition of Dust in the Lungs

The only dust that can be deposited in the alveolar region of the lung is that remaining in the inhaled air after passing through the respiratory tract (mucous-swept airways). Three physical mechanisms - impaction, gravity settlement, and diffusion - are instrumental in the deposition of dust in both the respiratory tract and the alveolar region. One property of a particle that is common to these three mechanisms is its 'aerodynamic size'. The aerodynamic size is defined as the diameter of a unit-density sphere having the same settling velocity as the particle in question.

The general dependence of alveolar and respiratory tract deposition on size and breathing rate is shown in Figure 1-1; however, the precise values vary with the breathing pattern and with the individual (1). There is, in general, a minimum deposition of 0.5-μm particles. Impaction and gravity-settlement deposition increase with increasing particle size and are responsible for most of the deposition at sizes above the minimum. Diffusion deposition increases with decreasing size and is the main mechanism below the 0.5-μm size.

In order to define the alveolar-deposition potential of an airborne dust cloud, the size distribution should be specified in terms of the aerodynamic size. Other measures of particle size may give misleading results; for example, coal particles with an aerodynamic size of 5 μm may have projected areas equivalent to those of 5- to 15-μm-diameter circles (2).

Clearance of Dust from the Alveolar Region of the Lungs

The mechanisms of clearance are not as clearly understood as are those of deposition. Experimental studies, reviewed by Hatch and Gross (1), have suggested that clearance is dependent on the composition, size distribution, shape, and concentration of the deposited dust.

Experimental studies (1) have suggestsd that the clearance mechanism has a half-life of 20 days or more, and only at extremely high dust concentrations has there been a suggestion of a breakdown in this mechanism. This slow rate of clearance suggests that the average dust exposure over a period of at least 20 days would be more important than peak exposures, unless they were extremely high.

Biologic Effect of Dust in the Lungs

The physical and chemical factors determining the biologic effects are: concentration, size distribution, composition, shape, and residence time. The parameters in which these factors should be expressed are not known. There is considerable support for the view (3, 4) that the mass of coal dust is the appropriate concentration parameter in coal miners' pneumoconiosis and that the surface area of silica dust is applicable to silicosis. However, some recent work (5) has suggested that a parameter intermediate to surface area and mass would be most closely related to the health hazard. For other materials and for mixed dusts, no recommendations have been made as yet. Therefore, in research applications, fairly comprehensive specifications of the dust clouds need to be made because of the uncertainty in both clearance and biologic effects.

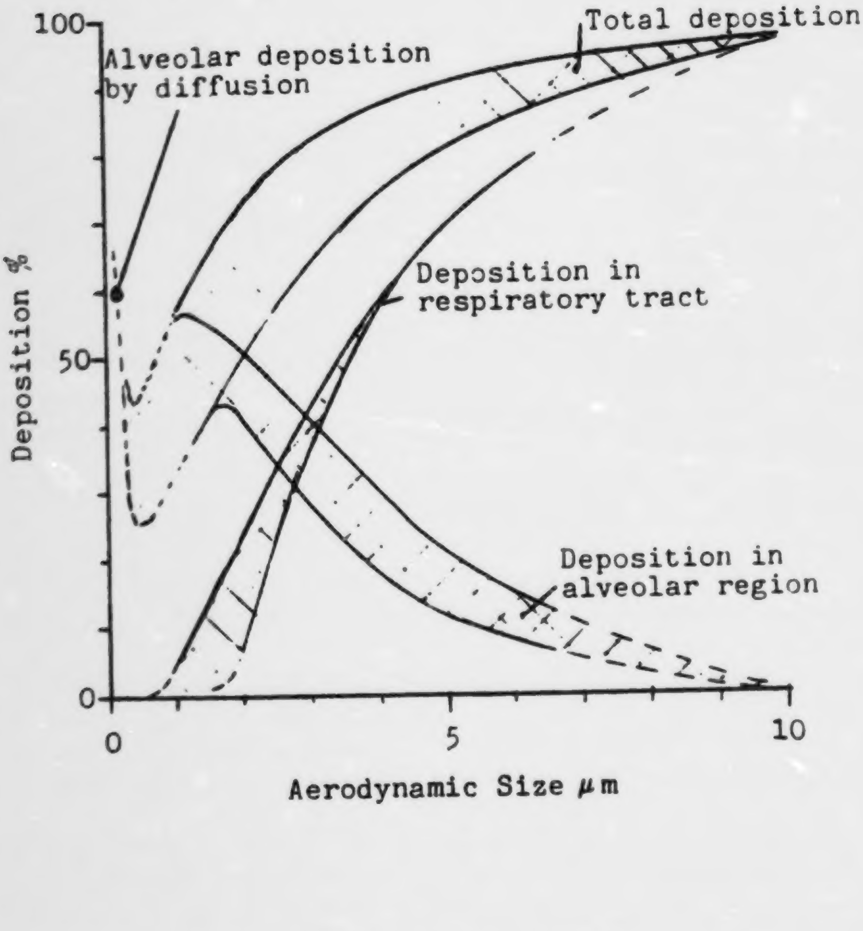

FIGURE 1.1 Dust deposition in the lungs showing range of variation with the breathing rate after Hatch and Gross (1).

SCOPE OF THIS PROJECT

Many comparisons of dust sampling instruments have been made in most parts of the world. Most of these comparisons have been restricted to a few working places or to laboratory dust chambers using just a few types of dust cloud. Per Odelycke (6) and Landwehr (7) have made two such studies, of wider range than most, which reveal very wide differences in the comparison of dust sampling instruments.

The object of the first part of this project was to investigate the range of dust concentrations obtained when a wide range of dust sampling instruments - in current and projected use - were exposed to a wide range of dust clouds prepared in the laboratory dust chamber. The dust clouds were prepared from five minerals and one synthetic material to cover the range of particle density from 1.4 to 5 gm/cm^3 and of particle shape from near cubic through plate to fibrous. The clouds were dispersed by five techniques to give a range of size distribution and different states of aggregation.

In these studies, particular attention was paid to the extent of variations - both random and systematic - in the relationship between systematic variations and measurable properties of the dust cloud. A number of subsidiary investigations are also reported which examined the collection efficiency of certain dust sampling instruments and compared aerodynamic respirable-dust size selectors.

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CHAPTER 2
EXPERIMENTAL TECHNIQUES

DUST CHAMBER

The laboratory dust chamber is 6x8x6 feet high inside. It is fitted with an inlet duct which distributes the incoming air evenly throughout the length of the chamber and with exhaust ducts. The chamber has an air circulating fan which has been arranged so that the dust laden air passes through the main 2x2x2-foot sampling space at low velocity (50 to 150 ft/min), through the fan, and around the walls to the sampling area again. The layout used in the main comparison experiment is shown in Figure 2-1. Most of the sampling instruments were placed, in the main sampling area, with their air inlets facing upstream.

DUST SAMPLING INSTRUMENTS

A number of important factors enter into the classification and description of dust sampling instruments, as follows:

1. property of the dust particles assessed - number, light scatter and mass;
2. method of collection - electrostatic or thermal precipitation, filtration, impaction, settlement, etc.
3. method of defining size range of dust assessed - instrument characteristics, assessment method characteristics, modification of instrument characteristics by fitting a primary dust collector to reject coarse non-respirable dust from the assessment;
4. method of assessment - weighing, microscopy, densitometry, tyndall effect, etc.

The range of instruments in this report covers the three properties and all the collection methods mentioned.

The methods of assessment used have been weighing for mass and microscope counting for number throughout. The tyndalloscope assesses the intensity of scattered light from the dust particles, and this is dependent on the surface area and the optical properties. Other methods for determination of mass and number have been used to a limited extent in parts of the world, i.e. the calibration of densitometric measurements of the Long Period Dust Sampler (1) in terms of both mass and number, by the National Coal Board, U.K., but have not been included in this study.

The classification of the instruments below follows the above list with the main sections corresponding to 1. The information given on each instrument comprises, a) manufacturer and description, b) installation in chamber and modifications, c) airflow and method of control, and d) method of assessment.

Figure 2-1 is an isometric representation of the position of the instruments in the chamber with indicating lines and crosses marking the projected position of each instrument air intake on the front wall of the chamber. The circulating fan is also shown.

The dust chamber is fitted with a 500 ℓ/min vacuum pump, to draw air through some of the filter type dust sampling instruments and the midget impingers.

To ease operation of the many dust sampling instruments, the vacuum pump and all the electrically operated instruments except the 113D dust sampler are switched on and off through a timer and relay.

Instruments Assessing Dust by Number

These instruments use three methods of dust collection: thermal precipitation, settlement, and impaction. Two types of instruments, the thermal precipitator and the konimeters, collected dust directly on glass slides which are later examined under the microscope. The impingers collect dust as a liquid suspension which is assessed by microscopic examination of settled dust.

Thermal Precipitators

The precipitators were made by Casella (2) Ltd., London, U.K., and are designated the T12500 thermal precipitator and the N.C.B. M.R.E. Long Period Dust Sampler Type 112A. The T12500 collects dust by thermal precipitation in a thin strip on two cover glasses. The type 112A is a modified thermal precipitator fitted with a battery operated air pump, inhaling and exhaling 2 cc of air through the sampling head every minute. The main modification is that the coarser respirable dust is permitted to settle on the glass cover slip prior to the thermal precipitation zone, thereby

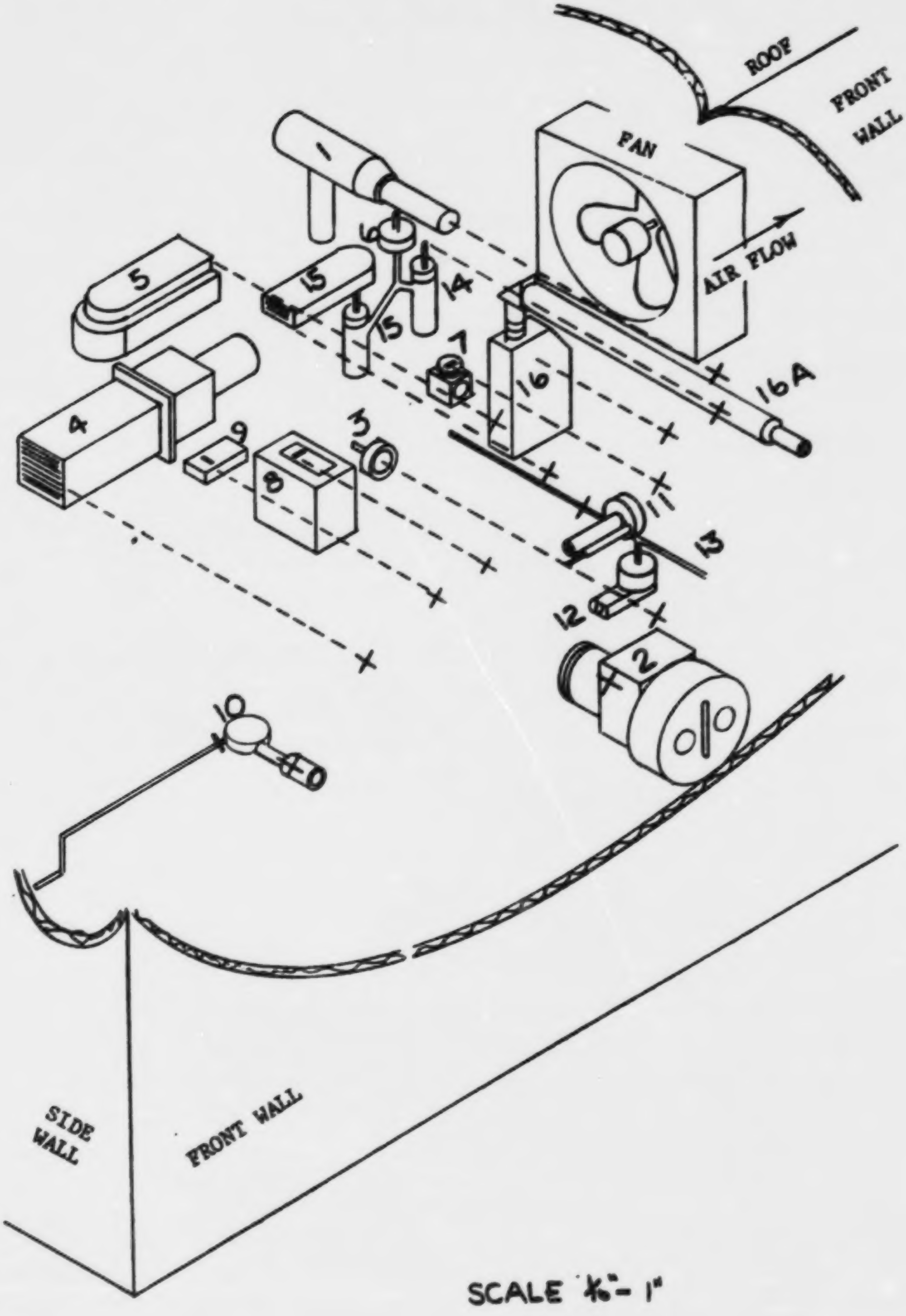
ROOF
FRONT WALL
FAN
AIR FLOW
1
6
5
15
14
15
7
16
16A
4
9
3
8
11
13
12
2
10
SIDE WALL
FRONT WALL
SCALE 1/10" = 1"

FIGURE 2-1: POSITION OF DUST SAMPLERS IN CHAMBER (ISOMETRIC PROJECTION)

Captions for Figure 2-1

Figure Reference	Dust Sampler
1	Electrostatic Precipitator
2	Open Filters - Staplex
3	Gelman
4	Respirable Dust Filters - Hexhlett
5	N. C. B. M. R. E. Type 103D
	(Note this sampler has, for clarity, been drawn 16 inches further from front wall)
6	Impinger Secondary Filter
7	Thermal Precipitators - Standard Thermal Precipitator, T 12500
8	- Long Period Dust Sampler, Type 112A
9	- Spare Head
10	Konimeters - Gathercole
11	Sartorius
12	Haslam
13	Suction Line
14	Impingers - Open Inlet
15	Fitted with Elutriator
16	Tyndalloscope
16A	Fitted with Remote Reading Telescope
X	Projected Position of Sampler Intakes on Front Wall

decreasing overlap and permitting eight hour samples to be collected. The sampler is also fitted with a horizontal elutriator (3, 4, 5) to remove the coarse, non-respirable dust. A spare 112A sampling head without the elutriator was also used.

The batteries were replaced by a battery charger operated from the mains with sufficient rheostats and ammeters to control the heater and motor currents.

The airflow of 2 cc/min through the Long Period Dust Sampler was obtained from its pump and calibrated by a bubble flowmeter. The airflow for the T12500 thermal precipitator was obtained from the standard aspirator and jet. The total volume of water displaced was measured for each run. The airflow of about 5 cc/min for the 112A spare head was obtained from a constant head aspirator, again the volume of water was measured for each run.

The thermal precipitator samples were counted using a light field projection microscope with a 2-mm oil immersion objective at 3000 times magnification in a number of size ranges:

a. greater than 5 µm by projected area*;
b. 1 - 5 µm*;
c. 0.5 - 1 µm;
d. all visible particles less than 0.5 µm**;
e. all visible fibres (fibres being defined as particles with a length breadth ratio greater than 3 to 1).

Midget Impingers

Three glass impingers (6) were used, one of which was fitted with a horizontal elutriator (2, 3) size selector to reject the non-respirable, coarse dust particles. The samples, after settlement for 30 minutes in 1-mm deep cells, were counted on a light field projection microscope with a 16-mm objective at a magnification of 1000 in two size ranges, greater and less than 5 µm*, and all visible fibres. The techniques used were those recommended by the United States Bureau of Mines (7) except that butyl alcohol was used to reduce evaporation and permit sampling times up to 2 hours. The impingers were selected to pass between 0.0975 and 0.1025 ft^3/min at 12-inch water gauge suction and the three together were operated with a back-up filter at 0.3 ft^3/min.

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* For the L. P. D. S. with a size selector, range a is included as an estimate of respirable dust in range b.

** This size range was counted only on two slides from each dust cloud using a light field binocular microscope fitted with a 1.3 N.A. oil immersion objective because the projection screen decreased the resolution slightly.

Konimeters

Three konimeters were used: Gathercole (Ontario), Haslam (South Africa), and Sartorius (Germany). The first two took 5 cm^3 snap samples while the third took both 2.5- and 5-cm^3 samples. These instruments, while basically the same, differ in size of and air velocity through the jet. These samples were counted, both before and after acid and heat treatment, at 160 times magnification on a dark-field microscope in two size ranges, greater and less than 5 µm, and all visible fibres. The preparation, heat treatment, and counting techniques used are those recommended by Mines Accident Prevention Association of Ontario (8). Comparisons with other instruments were made using the counts on untreated slides only.

The Haslam and Sartorius konimeters could not readily be placed in the chamber and operated remotely and they were set up to sample from tee pieces on a tube through which dust-laden air was drawn from the chamber continuously.

Instruments Assessing Dust by Light Scatter

The Leitz Tyndalloscope is the only instrument assessing dust by light scatter which is used to any extent in mining. The tyndalloscope (Leitz Germany, Model T II) was used. Non-respirable coarse particles were rejected by a settlement period of 20 seconds as recommended by the manufacturer. The instrument was modified for remote operation by fitting a telescope viewer and a rack and pinion drive. This modification required more elaborate calibration than the manufacturers suggest. This was done by using the Leitz intensity standard by itself and with each of the 0.7 and 1.0 density discs. These three calibration standards were cross-checked

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* On the impinger fitted with an elutriator, all particles were classified as being in the respirable size range and included with particles fewer than 5 µm.

with our instrument in normal operation and on a number of other tyndalloscopes, giving close agreement between all the instruments. The rack and pinion drive was then calibrated with all three intensity standards.
It is believed that these modifications may have reduced the sensitivity slightly but they did not affect the accuracy. The dust concentration is expressed as the square of the sine of the angle between the polarizing screens ($\sin^2 \theta$).

Instruments Assessing Dust by Mass

The mass of dust is greatly influenced by the coarse non-respirable particles in the dust cloud; therefore the instruments assessing dust by mass can be split into two classes: total-dust samplers and respirable-dust samplers with which size selectors (section below - Respirable Dust Size Selection) are used to remove coarse non-respirable particles.

Total-dust samplers - an electrostatic precipitator and open filters were used. The MSA (9) portable electrostatic precipitator collects dust on the inside of an aluminum tube. A number of open filters have been used at a wide range of flow rates. The dust was assessed by weighing and, in some cases, was dispersed in liquid for assessment by other techniques (Appendix A).

Respirable-dust samplers - three samplers, the Casella (2) Hexhlett (50 ℓ/min), the Casella Gravimetric sampler (2.5 ℓ/min), and a laboratory-build model (2.83 ℓ/min) with horizontal elutriator size selectors (MRC specification (3)) were used in the comparison with other instruments. In the work described in Chapter 5, a number of samplers fitted with cyclone and horizontal elutriator size selectors were compared with each other at various flow rates.

RESPIRABLE DUST SIZE SELECTION

Rejection of coarse non-respirable dust particles is included in most dust sampling techniques and is particularly important in gravimetric assessment.

The techniques available and where used are:

1. size assessment in the microscope - used in most number assessment techniques - 5-μm top size limit by projected area;

2. settlement chamber - a 20-second settlement period is recommended prior to using the tyndalloscope;

3. horizontal elutriators - the United Kingdom Medical Research Council produced a standard specification for respirable-dust size selection; horizontal elutriators to this standard specification have been used in the bulk of the work and were fitted to the Long Period Dust Sampler, one of the impingers, and all the respirable dust samplers used to obtain the results obtained in Chapter 3; horizontal elutriators to other specifications have been used to obtain some results given in Chapter 5;

4. cyclones - cyclone size selectors have been recommended because they are smaller, are independent of orientation and give a size selection curve closer to the average lung than does a horizontal elutriator. Comparisons between different models of cyclone size selectors (10, 11) are given in Chapter 5.

AIRFLOW CONTROL AND CALIBRATION

All sampling instruments with airflows of more than 1 ℓ/min have been calibrated against wet test gas meters either directly or through a tapered tube rotating float type airflow meter. The wet test gas meters used have been calibrated in a Fuels Division Laboratory in Ottawa.

The airflow control and monitoring, if any, used have either been that of the manufacturer or they have been added here. The instruments, in which no modifications to the manufacturer's airflow control and monitoring were made, are:

Electrostatic Precipitator,
Hexhlett (in the main experimental work, but was modified later - see Chapter 5),
Gravimetric Sampler,
T12500 Thermal Precipitator,
Long Period Dust Sampler,
Konimeter,
Tyndalloscope.

The Staplex was modified by reducing motor speed by voltage control and increasing the sensitivity of the airflow meter by reducing the area of the associated orifice.

The airflow for the other filters and the impingers was obtained by connection to a vacuum manifold through a control valve and a critical orifice (12) with a vacuum gauge on its upstream side. The airflow through a critical orifice (with a tapered kinetic to static pressure, recovery section) is dependent only on its area and the upstream pressure as long as the pressure drop across it is greater than 10% of the absolute pressure on its upstream side.

THE DUST CLOUDS

Comparative dust measurements were made in a large number of dust clouds chosen to study the effects of concentration, size distribution, density, shape, and aggregation. Some subsidiary experiments on collection efficiency were carried out in dust clouds prepared by a compressed air atomizer from a dilute latex of uniform sized plastic spheres.

The effect of concentration was examined by preparing three to six dust clouds of each of twenty-two types covering a concentration range of between 4 to 1 and 10 to 1; the lowest concentration used was high enough to avoid excessive contamination by atmospheric dust. Some further comparisons were made at lower concentrations in three types of dust cloud after fitting a filter to the chamber air inlet.

Dust clouds differing in size distribution were prepared from each material by four dry methods of dust dispersion:

1. laboratory twin-jet fluid-energy mill (13),
2. rotating plate pulverizer (14),
3. compressed air ejector followed by a cyclone,
4. as 3, but bypassing the cyclone.

The feed material for methods 1 and 2 was fairly coarse, larger than 200 mesh, but that for methods 3 and 4 was prepared from coarse material by breaking in the rotating plate pulverizer. The dust clouds prepared by methods 3 and 4 from glass fibre and asbestos were not used because they contained very low concentrations of fibrous particles.

Dust clouds were prepared from coal, silica, and pyrite to study the effect of particle density; these clouds consisted of particles of near cubical shape. To study shape, clouds prepared from the minerals silica, mica, and asbestos were used. Because many of the asbestos fibres were very fine and could not be resolved in an optical microscope, glass fibre (diameter 0.25 - 0.50 μm) dust clouds were also examined.

Two types of aggregated coal dust clouds were prepared by using a spinning disc sprayer to atomize two strengths (full strength - 0.11 gm/ml, 1.4×10^{10} particles/ml; dilute - 1/10 full strength) of suspensions of coal dust in ethyl alcohol. The coal dust was prepared in the jet mill. Attempts to prepare aggregated silica dust clouds by a similar technique were unsuccessful. The preparation and properties of the dust clouds are described in detail in Appendix A.

TECHNIQUES FOR ANALYSIS OF RESULTS

The analysis is described in detail in Appendix B.

It can be expected that the relation between the dust concentrations measured by two types of instrument in one type of dust cloud should be given by the equation,

$$y = kx \qquad \text{(Eq. 1)}$$

where y and x are the dust concentrations measured by the two instruments and k is a constant.

The value of k can be expected to differ in the various types of dust clouds; this can be allowed for by using dummy variables (15). Then,

$$y = x(1+k_1 x_1)(1+k_2 x_2) \ldots (1+k_n x_n) \qquad \text{(Eq. 2)}$$

where x_2 to x_n are the dummy variables taking the value $x_i = 1$ if the measurement is made in the ith dust cloud and $x_i = 0$ if it is not. k_1 to k_n are simply related to the coefficients in each type of dust cloud as shown by reducing equation 2 to fit one dust cloud,

$$y = x(1 + k_1 . 1) \qquad \text{(Eq. 3)}$$

All the other brackets reduce to 1 as, $x_2 = x_3 = \ldots = x_n = 0$.

In order to perform a regression analysis and meet the requirements for a valid statistical analysis based on least squares, it is necessary to transform this equation to a more suitable form. The most suitable form was found to be,

$$\ln \frac{y}{x} = b_0 + b_2 x_2 + \ldots + b_n x_n \qquad \text{(Eq. 4)}$$

where $\ln \frac{y}{x}$ is the dependent variable,
x_2 to x_n are the independent variables, and
b_0 to b_n are the regression coefficients.

This equation was solved using a computer with the IBM program REGRE (16).

In solving this equation it was found that checks for linearity could readily be made. Those checked showed that all relationships were linear except those between the konimeters and the other instruments. In these cases the modified relationship was used.

$$\ln \frac{k^S}{x} = b_0 + b_2 x_2 + \ldots + b_n x_n \qquad \text{(Eq. 4a)}$$

where k is the dust concentration measured by the konimeter and
S is a coefficient of non-linearity determined by the methods given in Appendix B.

The value of the coefficient S in the relationship between x and y is first determined by methods given in Appendix B. Only in comparisons with the konimeters was S found to differ significantly from unity.

The computer program was used to study the relationship between each pair of instrument types (for example, one type being the four konimeters). In this, values were given for:

1. the geometric mean ratio between the dust concentrations estimated by a pair of instrument types in all the dust clouds;

2. the difference between the mean ratio in each type of dust cloud and the overall mean;

3. the standard error of 2 above;

4. the linearity of 1 and 2 with dust concentration.

The computer program can be used to compare the dust concentration estimated by the individual instruments of one type with its mean; this gives:

5. the mean differences in the estimates of the dust concentration by the two to four instruments of each type;

6. the standard error of a dust concentration estimate by a single instrument.

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CHAPTER 3
COMPARISONS BETWEEN INSTRUMENTS ASSESSING DUST BY NUMBER OF PARTICLES

All dust concentrations by number are expressed as particles per cubic centimetre (p/cm^3).

The results presented in this chapter are abstracted and analysed to demonstrate the conclusions that have been drawn from this work. The detailed results of each test are given in Appendix C.

This section describes the results obtained in comparisons made between instruments assessing dust by number.

A typical comparison between two sampling instruments of different types is shown in Figure 3-1. This shows the wide range of results obtained, for instance, in dust clouds with concentrations estimated at 1000 p/cm^3 by a konimeter; the impinger estimates the concentration to be 100 to 500 p/cm^3. The range would be even wider if the results obtained in fibrous dust clouds had been included.

ACCURACY OF DUST ESTIMATES

The computer analysis of the dust measurements made within each group of instruments calculated the departure of the concentration estimated by the individual instruments from the group mean and the standard error for a single observation. The results obtained in the coal, silica, pyrite, and mica dust clouds are shown in Table 3-1.

The mean difference between instruments in the same group is due mainly to differences in collection efficiency and to systematic counting errors. The first probably accounts for most of the differences between the Haslam, Gathercole, and Sartorius 5-cm^3 sample konimeters, while the second accounts for the low estimates on the standard thermal precipitator as compared with the long period dust samplers and on the Sartorius 5 cm^3 as compared with the 2.5 cm^3. The konimeters give less reproducible dust concentration estimates than do the impingers and thermal precipitators.

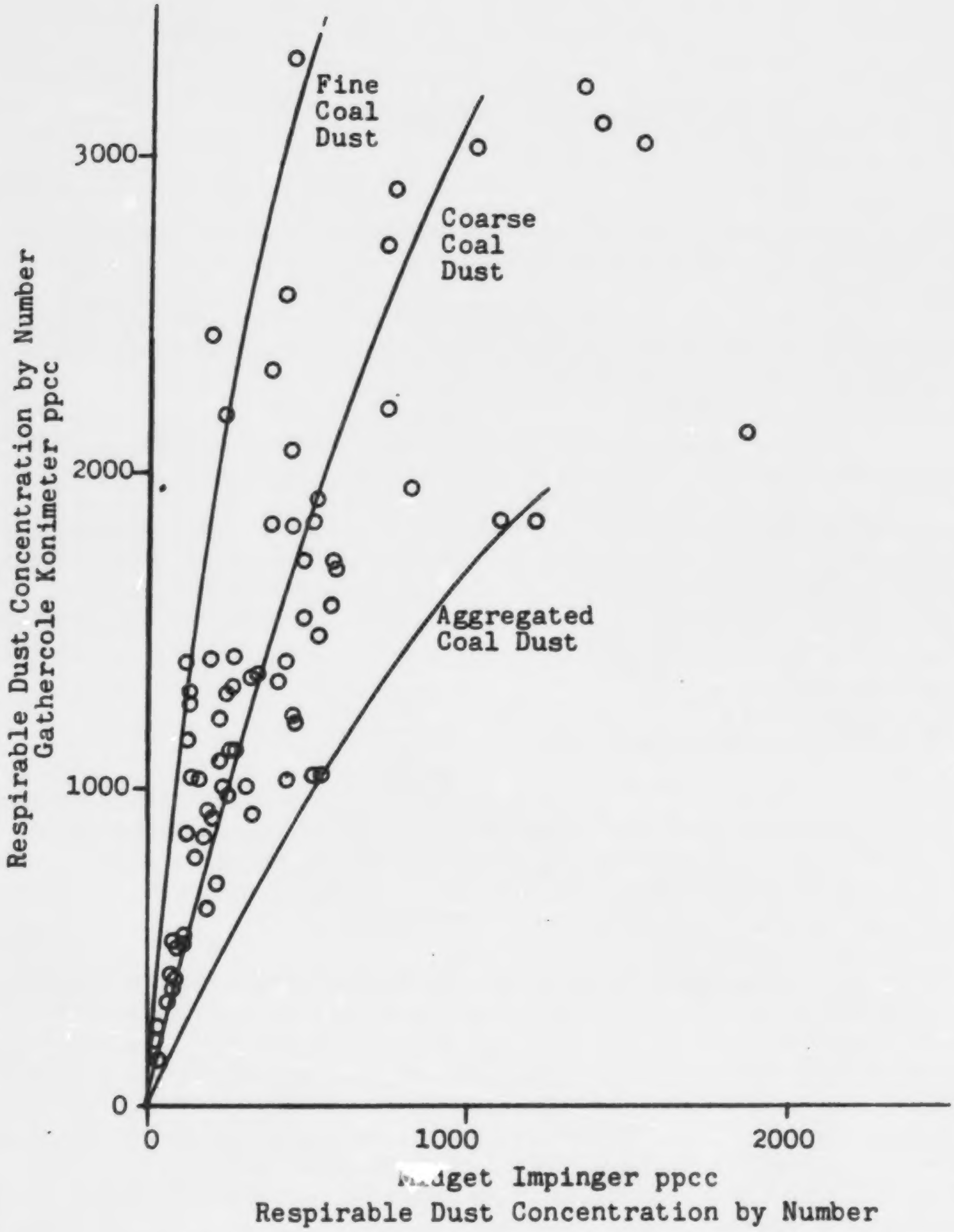

FIGURE 3.1 COMPARISON BETWEEN INSTRUMENTS ASSESSING DUST BY NUMBER with 'best fit' lines by statistical regression.

TABLE 3-1

Accuracy of Dust Estimates

Group Instrument	Ratio of Individual Instrument to Group Mean	Geometric Standard Error for Single Observation
Thermal Precipitators 1-5 µm Size Range		1.24
Standard	0.89	
Long Period (Head Only)	1.03	
Long Period	1.06	
Thermal Precipitators 0.5-5 µm Size Range		1.21
Standard	0.91	
Long Period (Head Only)	1.01	
Long Period	1.06	
Impingers		1.19
1	1.03	
2	0.98	
Elutriated	0.99	
Konimeters		1.32*
Haslam	0.84	
Gathercole	0.93	
Sartorius 5 cc	1.0	
2.5 cc	1.21	

* This is for the mean of five samples taken in each run. Normal practice is to take the mean of three samples.

THE INSTRUMENT COMPARISON INDICES

The instrument comparison indices are the geometric mean ratios of the dust concentrations measured by a pair of instruments in one type of dust cloud. The values for the comparisons between the number counting instruments are shown in Table 3-2, together with the range of the standard error of the index in each of the coal, silica, pyrite, and mica dust cloud types. It can be seen that the range of each comparison index is very high - 3.7 to 8.4 in the coal, silica, pyrite, and mica clouds - reaching 250 to 1 when the fibrous dust clouds are included. The differences in the index between dust clouds are mostly much larger than the standard error of the index in any one cloud.

THE EFFECT OF CONCENTRATION

The main comparison was carried out over a range of concentrations between 4 to 1 and 10 to 1 in each type of dust cloud. The non-linearity in the comparisons between each pair of instruments was examined by comparing the coefficients b_1 and $1/b_1'$ in the two regression equations:

$$\ln y = b_0 + b_1 \ln x + b_2 X_2 + \ldots + b_n X_n$$

and

$$\ln x = b_0' + b_1' \ln y + b_2' X_2 + \ldots + b_n' X_n$$

as described in Appendix B. It was found that this coefficient had a statistically significant value only in the comparisons with the konimeters. The relationships between the other instruments can thus be assumed linear.

The Non-Linearity of the Comparisons with the Konimeter

The regression analysis carried out on the main set of experiments showed that the comparisons between the konimeters and the other instruments were not linear with concentration. The regression analysis can be used to produce a power law of the form:

$$y = aK^S$$

where y is the dust concentration measured by another instrument; K is that

TABLE 3-2

Instrument Comparison Indices: Number/Number

Material and Dispersion Method	Instrument Comparison Index (Non-dimensional)				
	TP* 1-5 µm / Impinger	TP* ½-5 µm / Impinger	TP* 1-5 µm / Konimeter	TP* ½-5 µm / Konimeter	Impinger / Konimeter
Coal					
Aggregated					
Full Strength	0.51+	0.83+	0.34	0.56	0.67+
Dilute	0.85+	1.74+	0.36	0.74	0.42+
Jet Milled	1.75	5.5	0.19	0.65	0.115
Pulverised	2.3	6.2	0.30	0.88	0.14
Cyclone	1.8	3.2	0.42	0.74	0.22
No Cyclone	1.7	3.1	0.52	0.90	0.28
Silica					
Jet Milled	1.5	4.8	0.27	0.91	0.19
Pulverised	1.25	3.8	0.32	0.99	0.25
Cyclone	1.26	2.7	0.39	0.83	0.30
No Cyclone	1.28	2.8	0.32	0.72	0.24
Pyrite					
Jet Milled	0.62	5.25	0.13	1.12	0.24
Pulverised	1.03	6.95	0.30	2.05	0.28
Cyclone	1.04	2.0	0.32	0.68	0.31
No Cyclone	.99	1.9	0.30	0.63	0.31
Mica					
Jet Milled	1.26	3.7	0.28	0.86	0.255
Pulverised	1.18	4.9	0.19	0.83	0.175
Cyclone	0.93	3.0	0.22	0.80	0.27
No Cyclone	0.96	3.1	0.22	0.74	0.26
Geometric Standard Error Max.	1.18	1.28	1.16	1.18	1.22
Min.	1.17	1.15	1.14	1.14	1.15
	Non Fibrous and Fibrous Particles				
Glass Fibre					
Jet Milled		1.95		0.24	0.088
Pulverised		5.7		0.28	0.051
Asbestos					
Jet Milled		4.7		0.47	0.095
Pulverised		3.3		0.51	0.155
	Fibrous Particles Only				
Glass Fibre					
Jet Milled		0.62		2.1	2.95
Pulverised		2.1		3.9	1.22
Asbestos					
Jet Milled		7.3		63	10.1
Pulverised		4.25		15.7	3.0

* Thermal precipitator with size range.

+ These results exclude the impinger fitted with an elutriator (see Section 5.8).

estimated by the konimeter; a is a constant dependent on the cloud type; and S is the exponent. The best values of the exponent are between 1.1 and 1.4 for comparisons with the four konimeters in the main set of experiments.

However, this equation is only valid over a limited range of concentrations and an alternative presentation of the results obtained in three types of dust cloud is shown in Figure 3.2. This figure shows the dependence of the konimeter comparison index* on the dust concentration. It appears that four major phenomena occur:

1. an apparent difference in the type of relationship for the Haslam konimeter and the others;
2. a sharp increase in the instrument comparison index at low concentrations on three of the four konimeters;
3. a gradual decrease in the index at concentrations by mass above a few milligrams per cubic meter;
4. a marked difference in the comparison index with some konimeters in some dust clouds between the main set of experiments and the later set; this appears to be akin to the 'wandering bias' described by Beadle (1).

The reasons for these phenomena are not clear. Beadle (1) has described non-linear and erratic behaviour of konimeters. The Haslam konimeter clearly differs from the Gathercole and Sartorius konimeters in having a much weaker pump action.

THE EFFECT OF PARTICLE SIZE DISTRIBUTION

Data on the size distributions of the dust clouds determined by a number of techniques are given in Appendix A. These have been used as size classifications and a correlation analysis with the value of each comparison index was carried out. It was found that the comparisons between all these instruments showed a significant correlation with one or more of the size classifications.

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* The comparisons were made with respirable mass (mg/m^3) as the Hexhlett gave the most reproducible estimate of concentration over such a wide range.

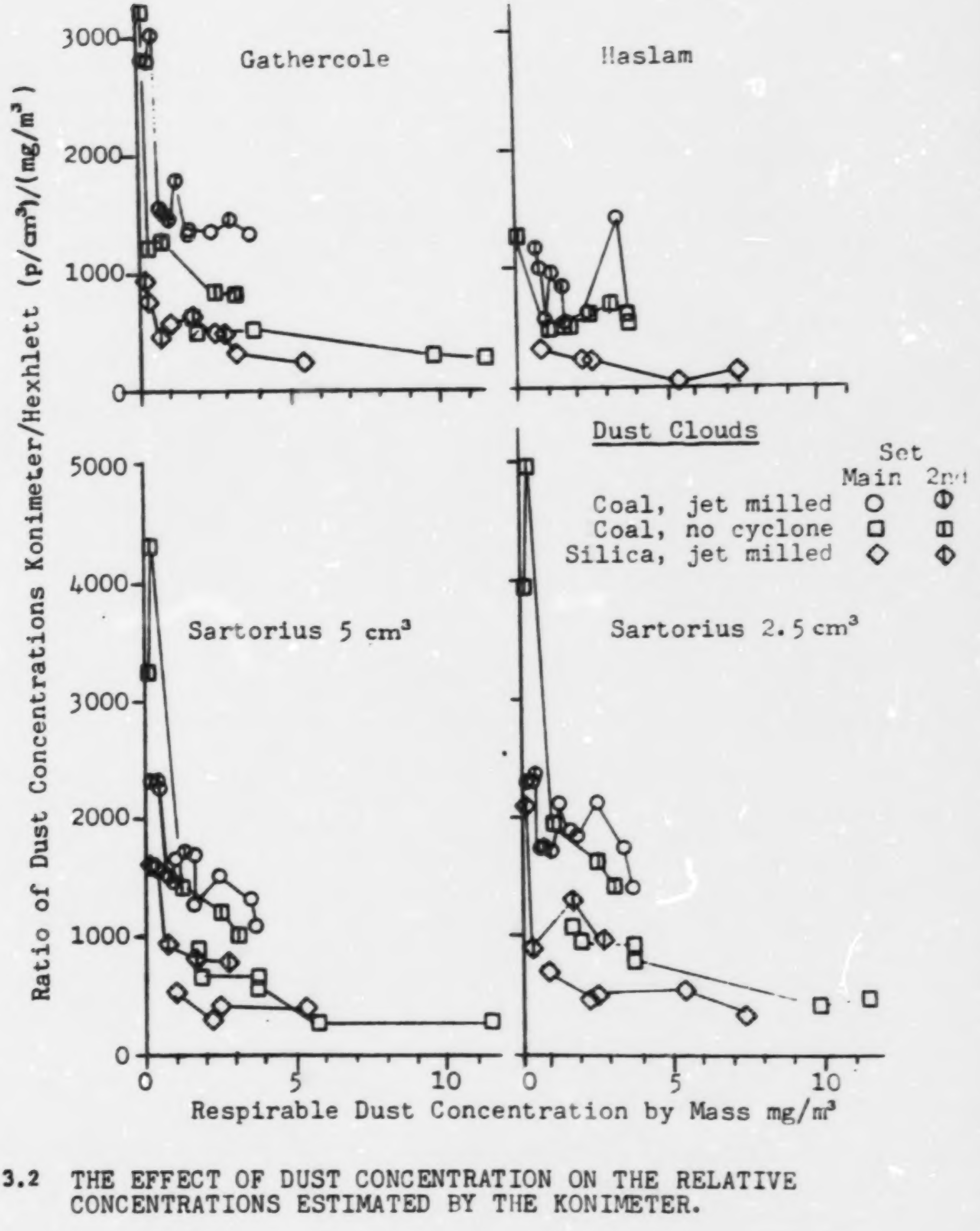


FIGURE 3.2 THE EFFECT OF DUST CONCENTRATION ON THE RELATIVE CONCENTRATIONS ESTIMATED BY THE KONIMETER.

The comparison indices are shown in Figure 3.3, plotted against the size classification by projected area obtained by microscopic examination of the thermal precipitator samples. It can be seen that there are apparent marked changes in index with size distribution in most of these comparisons. It can be seen that the concentration estimates made by the impingers and konimeters in comparison with the thermal precipitators (1 to 5-μm size range) tend to fall in the coarser dust clouds, while in comparison with the 0.5to 5-μm size range they tend to rise.

EFFECT OF DENSITY

The materials, coal, silica, and pyrite, were chosen to cover a range of densities with particles of similar near-cubic shape. There are suggestions of differences between these three materials as can be seen in Figure 3.3, particularly in the comparisons with the impinger. It appears that the relative count by the impinger increases with density in the order, coal (density 1.4 g/cm^3), silica (2.6 g/cm^3), and pyrite (5 g/cm^3).

EFFECT OF PARTICLE SHAPE

The materials, silica, mica, glass fibre, and asbestos, were chosen to give particles of different shapes; near cubic, plate, coarse fibre and fine fibre respectively. Most of the glass fibres were 0.25 to 0.5 μm in diameter but many of the asbestos fibres could not be resolved.

Only minor differences were found between silica and mica, as shown in Figure 3.3. Extreme values of the comparison index were found in some of the fibrous dust clouds, particularly in the assessment of fibrous particles alone (Table 3.2). The 'shape' effect cannot be distinguished from possible size distribution effects because the size distributions of the fibrous dust clouds differ greatly from those of the other dust clouds. The fibrous dust clouds have three components; fine particles less than 1 μm, fine fibres, and a coarse non-respirable portion of matted fibres, while there are very few particles of intermediate size. The other dusts have a smooth distribution from 0.5 to 10 μm.

EFFECT OF AGGREGATION

Dust clouds containing aggregated particles were prepared by spraying coal dust suspensions; the particles in each liquid drop remained attached to each other after evaporation. Two types of cloud were prepared from full strength and dilute suspension respectively. The

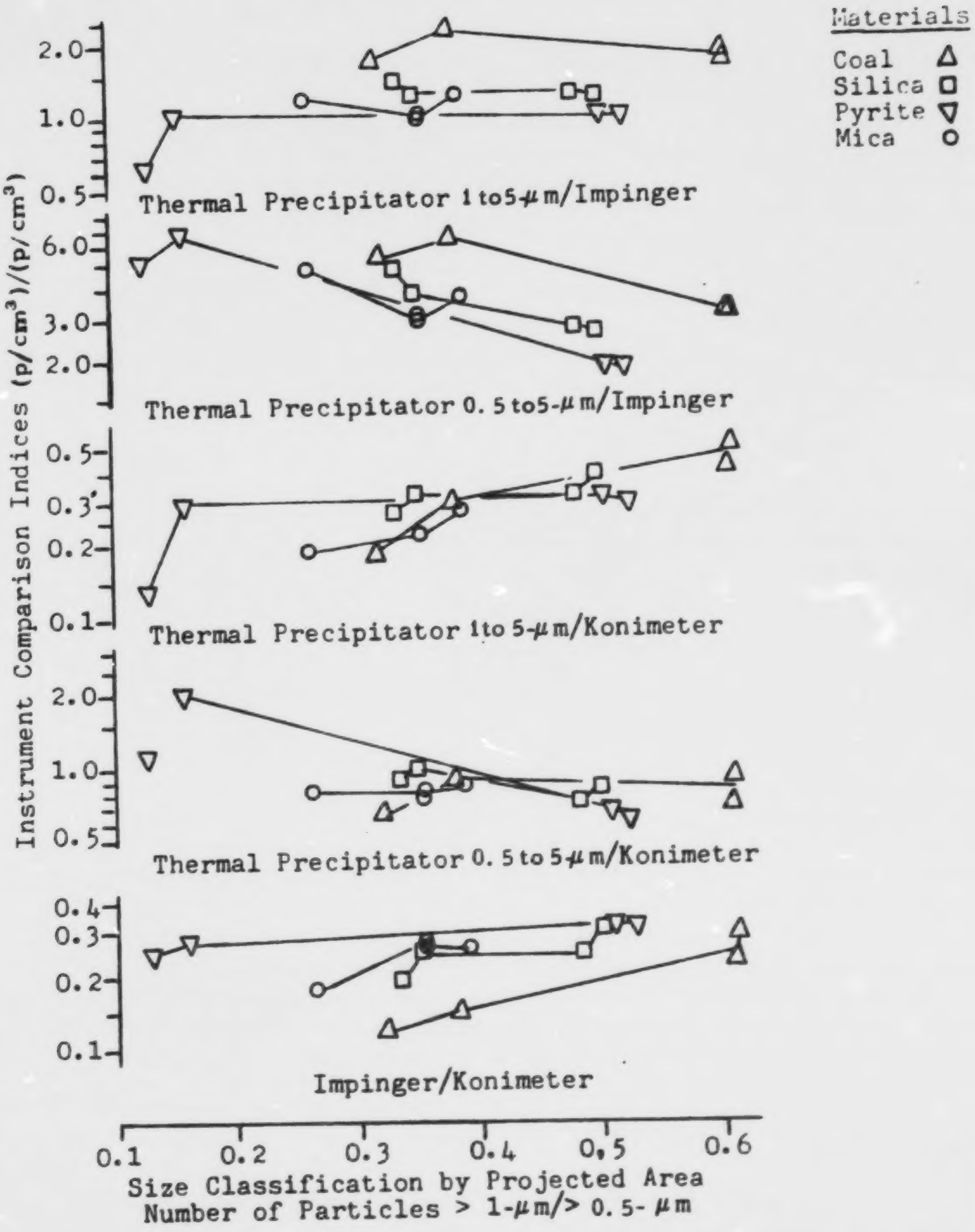


FIGURE 3.3 EFFECT OF PARTICLE SIZE AND DENSITY ON COMPARISON INDICES OF DUST SAMPLING INSTRUMENTS.

distribution of particles among the aggregates is shown in Table 3-3. It was not possible to distinguish particles and identify aggregates in the 0.5 to 1-µm size range. In contrast, there were very few aggregates in the coal, silica, pyrite, and mica dust clouds prepared by dry dispersion. The number concentration for the thermal precipitator samples was obtained by following the NCB rule of one count for each set of touching particles.

The instrument comparison indices for the aggregated dust clouds were given in Table 3.2. The index in the aggregated dusts extends outside the range of those for the other non-fibrous dust clouds for the comparisons with the impinger and for the comparison between the thermal precipitator (0.5 to 5-µm size range) and the konimeter.

TABLE 3-3

Distribution of Particles in Aggregated Dust Clouds

	Individual Particles %	Relative Number of Aggregates Containing 2 or 3 Particles %	4 or more Particles %
Full strength suspension			
> 5-µm size range	47	40	13
1 to 5-µm size range	80	20	0.3
Dilute suspension			
> 5-µm size range	52	30	18
1 to 5-µm size range	81	19	.1

The midget impingers gave relatively high counts because the dust particles forming the aggregates are dispersed in the collecting liquid and counted separately. The impinger, fitted with an elutriator, gave a count 0.7 times those of the other two impingers, suggesting that the coarse non-respirable aggregates contained a sufficient number of small particles to increase the count by 40%.

COMPARISON OF MICROSCOPE TECHNIQUES

The wide variations in the instrument comparison index, particularly in the fibrous clouds, led to a minor study in which two thermal precipitator (Long Period Dust Sampler) slides from each type of dust cloud were counted using the impinger and konimeter microscope assessment techniques. The geometric means of the ratios from each pair of slides are shown in Table 3.4 as the microscope comparison index. These results show considerable scatter; the T.P./Impinger microscope comparison has a range of 7 to 1, the T.P./Konimeter 6.7 to 1, and the Impinger/Konimeter 4.3 to 1. The most extreme values occur in the finest dust clouds, jet-milled pyrite and asbestos fibres. Apparently neither the impinger microscope technique with its medium-resolution light field nor the konimeter microscope with its medium resolution dark field see or count particles less than 0.5 µm in size. Also the impinger and konimeter microscope techniques are not nearly as effective as the high-resolution thermal precipitator microscope in distinguishing fine fibres.

COMPARISON OF THE COLLECTION EFFICIENCIES

The collection efficiency of the instruments was examined using three techniques:

1. in dust clouds prepared from uniform-sized spheres;
2. by using the same microscope technique on samples taken by the different techniques;
3. for impingers only, weighing the dust in the air passing through the impingers after collection on a filter.

The clouds of uniform-sized spheres were prepared by atomizing with compressed air a dilute aqueous latex (Dow Chemical Co.) of polystyrene spheres. The size used is that given by the maker and measured by electron microscopy; however, the surface active agent used to disperse the spheres is hygroscopic and forms a thin liquid film over each sphere. It is believed that the film is less than 0.1 µm thick, i.e. the diameter of the spheres is between the stated size, d, and d + 0.2 µm. Because high-resolution microscopy is needed to distinguish and count the finer spheres, 0.2-mm settling cells were used for the impinger and the konimeter spots were collected on cover slips. Because the density of the spheres, about 1.0 g/ml, was not much greater than that of the alcohol, 0.9, the settling periods in the impinger cells were increased to permit full settlement.

TABLE 3-4

Comparison of Microscope Techniques on Thermal Precipitator Slides

Dust Cloud	Microscope Comparison Index		
	T.P.* / Impinger	T.P.* / Konimeter	Impinger / Konimeter
Coal			
Jet Milled	1.3	1.35	1.05
Pulverised	1.5	1.15	0.75
Cyclone	1.55	1.0	0.67
No Cyclone	1.25	1.4	1.1
Silica			
Jet Milled	1.6	1.4	0.88
Pulverised	1.7	1.2	0.71
Cyclone	1.3	1.0	0.80
No Cyclone	1.4	1.1	0.63
Pyrite			
Jet Milled	0.84	2.9	3.8
Pulverised	1.6	1.5	0.86
Cyclone	1.3	1.1	0.85
No Cyclone	1.0	1.0	1.0
Mica			
Jet Milled	1.55	1.4	0.9
Pulverised	1.4	1.0	0.75
Cyclone	2.3	1.1	0.53
No Cyclone	1.8	1.45	0.98
	Non Fibrous and Fibrous		
Glass Fibre			
Jet Milled	1.15	1.5	0.96
Pulverised	1.4	1.1	1.0
Asbestos			
Jet Milled	3.9	2.4	0.62
Pulverised	2.7	3.2	0.80
	Fibres Only		
Glass Fibre			
Jet Milled	1.6	3.75	2.3
Pulverised	3.0	6.7	2.2
Asbestos			
Jet Milled	6.0	3.8	0.87
Pulverised	2.4	4.5	1.75

* The thermal precipitator technique counts are in the 0.5 to 5-µm size range and/or all visible fibres.

The results obtained are summarized in Figure 3.4. While it is generally (2) accepted that thermal precipitators have a very high efficiency, these results show that they collect less than do the impingers in the 1 to 2-µm size range. This is believed to be due to the deposition of some of the spheres in the metal intake channels of the thermal precipitators due to an electrostatic charge on the spheres, charge separation having occurred during atomization. Efforts to neutralize the charges with an ionizer were only partly successful.

Figure 3.4 shows that the comparative collection efficiency of the impingers falls rapidly for spheres less than 1 µm in diameter. The konimeters, with the normal adhesive layer (prepared from 2% petroleum jelly in xylene), show low collection efficiencies for spheres of all sizes. The spheres were also spread over an area much larger than the normal spot, suggesting that bouncing occurred. However, when using a thick adhesive layer (10% petroleum jelly), the spheres were only found in the normal spot area and the collection efficiencies were much higher, rising to slightly more than that of the thermal precipitators. A steep fall in collection occurred below 0.7 µm on the Gathercole and Sartorius konimeters but for the Haslam, with its much softer action, the fall occurred between the 1.8 and 1.3-µm-diameter spheres.

In the second technique, the results obtained by normal counting of impinger and konimeter samples were compared with special counts of the thermal precipitator samples using the impinger and konimeter microscope techniques. This technique, of course, compares the thermal precipitator with the impinger and its associated settling and counting cell. The results obtained are shown in Table 3-5. It can be seen that the thermal precipitator collects 1.2 to 7.5 times more dust than does the impinger, the effect being particularly noticeable in the fine dust clouds - jet-milled pyrite and fibres. Thus, the lower size limit is defined by the collection and settlement characteristics of the impinger and its cell. The konimeter gives a much higher count, up to five times except for fibres only, than the thermal precipitator; this again is most marked in the clouds containing fine particles. As the thermal precipitator is reputed to have a very high collection efficiency, these results suggest that the konimeter can produce spurious particles, or countable impressions, in the adhesive layer with fine dust particles. It is clear that the impinger and the konimeter are poor at collecting fine fibrous particles.

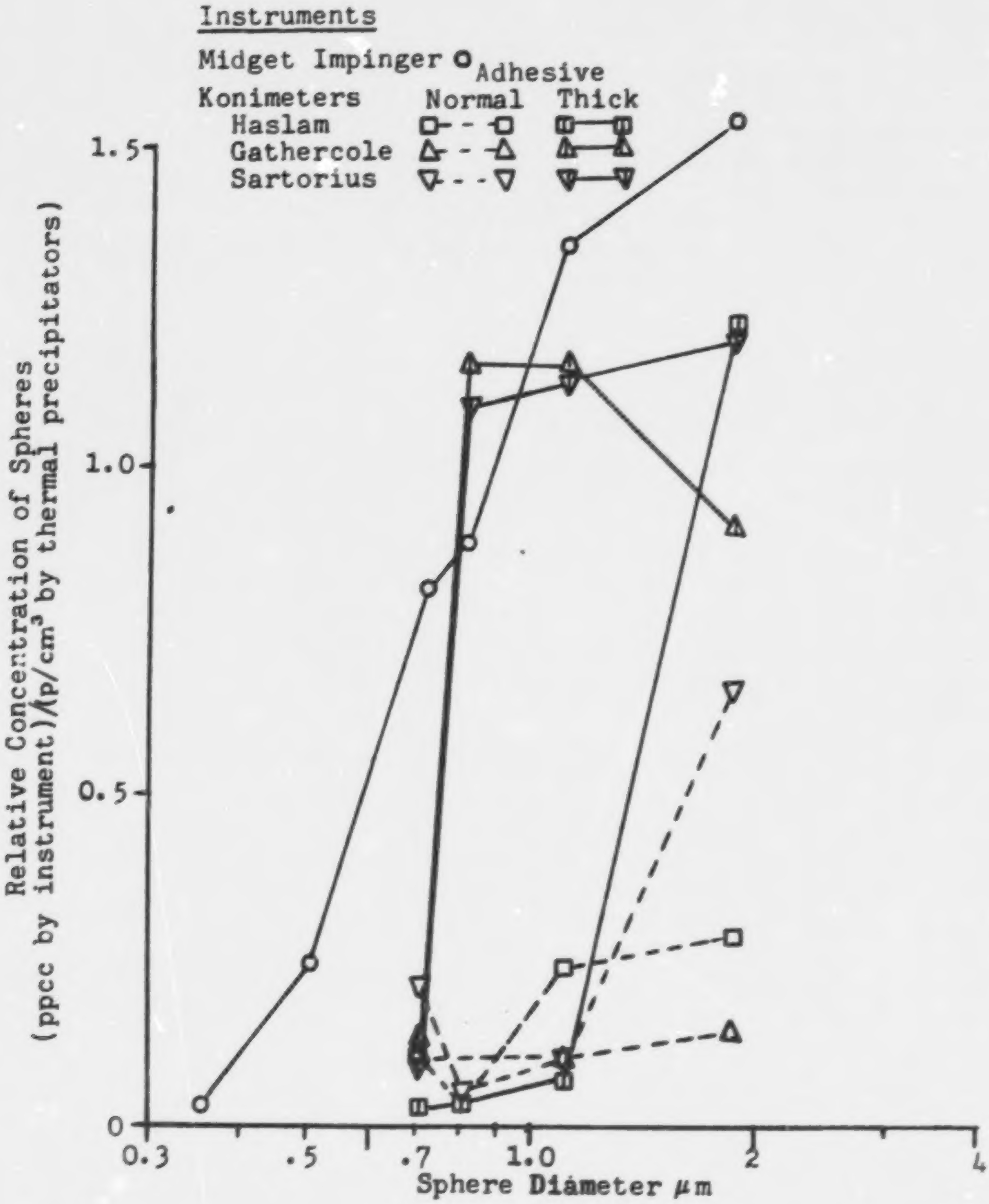


FIGURE 3.4 COLLECTION EFFICIENCIES OF IMPINGERS AND KONIMETERS COMPARED TO THAT OF THERMAL PRECIPITATORS.

TABLE 3-5

Comparison of Thermal Precipitators with the Impingers and Konimeters using the Impinger and Konimeter Microscope Techniques Respectively

Dust Cloud	Collection Comparison Index	
	T.P.* / Impinger**	T.P.+ / Konimeter
Coal		
Jet Milled	3.7	0.39
Pulverised	5.5	0.79
Cyclone	2.4	0.56
No Cyclone	3.1	0.62
Silica		
Jet Milled	3.9	0.74
Pulverised	2.45	0.64
Cyclone	2.05	0.6
No Cyclone	1.7	0.44
Pyrite		
Jet Milled	5.4	0.23
Pulverised	5.0	1.05
Cyclone	2.6	0.53
No Cyclone	1.75	0.46
Mica		
Jet Milled	1.7	0.48
Pulverised	3.9	0.89
Cyclone	1.2	0.53
No Cyclone	1.5	0.46
	Non Fibrous and Fibrous	
Glass Fibre		
Jet Milled	2.65	0.2
Pulverised	5.4	0.26
Asbestos		
Jet Milled	1.25	0.21
Pulverised	1.5	0.19
	Fibres Only	
Glass Fibre		
Jet Milled	2.3	4.2
Pulverised	4.1	2.3
Asbestos		
Jet Milled	3.1	61
Pulverised	7.5	33

* Thermal precipitator assessed by impinger microscope.

+ Thermal precipitator assessed by konimeter microscope.

** Includes effects due to both collection and counting cell settlement.

In the third technique the filter. placed in the exhaust air from the impingers, collected between 4 and 20% of the mass of respirable dust (Hexhlett) present in the dust cloud, i. e., the collection efficiency of the impingers was 80 to 96% by mass in the respirable size range. The low values occurred in the fine dust clouds and the high in the coarse.

THE COMPARISON OF COUNTING SIZE RANGES ON THERMAL PRECIPITATOR SAMPLES

There is general agreement throughout the world that the top limiting size of respirable dust is 5 µm and that particles with a projected area larger than a 5-µm-diameter circle are not counted; however, widely different lower limiting sizes have been used: 1 µm, 0. 5 µm and all visible (A. V.) particles with various microscope techniques. Table 3-6 shows the relative increase in number concentrations that are found with the thermal precipitator when the 1to5-µm counting range is extended to finer sizes.

THE EFFECT OF HEAT TREATMENT AND ACID WASH ON KONIMETER DUST ESTIMATES

The incineration and acid wash treatment of konimeter slides was introduced in South African gold mines to emphasize the silica content in dust assessment, and the treatment is also recommended by the MAPAO (3) for use in high-silica mines in Ontario. The counting of konimeter samples in this study was done both before and after treatment. All the results given elsewhere in this report refer to counts on untreated slides. The effect of treatment expressed as the mean ratio of treated to untreated counts for each material was 0. 05 for coal, 1. 04 for silica, 0. 91 for pyrite, 0. 96 for mica, 0. 61 for glass fibre, and 0. 41 for asbestos. The variability of these ratios was high, the standard error of a single observation (mean of 5 spots) being about 70%.

REFERENCES

1. Beadle, D. G. , "An Investigation of the Performance and Limitations of the Konimeters", J. Chem. Met. Min. Soc. S. A. , Vol. 51, pp 265-283 (1951).

2. Anon. , "Air Sampling Instruments for Evaluation of Atmospheric Contaminants", Amer. Conf. of Gov. Ind. Hyg. , p. B-5-3 (1966).

TABLE 3-6

Comparison of Counting Size Range on Thermal Precipitator Slides

Dust Cloud	Counting Size Range Comparison Index	
	$\frac{0.5 - 5\ \mu m}{1 - 5\ \mu m}$	$\frac{A.V.^{*} - 5\ \mu m}{1 - 5\ \mu m}$
Coal		
Jet Milled	3.1	7.2
Pulverised	2.9	6.2
Cyclone	1.8	2.5
No Cyclone	1.8	---
Silica		
Jet Milled	3.3	12
Pulverised	2.7	3.5
Cyclone	1.6	2.4
No Cyclone	2.1	2.6
Pyrite		
Jet Milled	8.9	23
Pulverised	5.7	35
Cyclone	2.2	3.0
No Cyclone	2.05	---
Mica		
Jet Milled	3.1	16
Pulverised	5.5	16
Cyclone	3.3	5.5
No Cyclone	3.15	---

* All particles visible using a high-resolution microscope (2 mm oil immersion, light field, N.A. ≃ 1.0) were counted.

3. Anon, "Konimeter Dust Sampling as Practised by MAPAO Engineers", Mines Accident Prevention Association of Ontario (1959).

CHAPTER 4
COMPARISON BETWEEN INSTRUMENTS ASSESSING DUST BY NUMBER, BY LIGHT SCATTER AND BY MASS

This chapter summarizes the comparisons between instruments assessing dust by number, by light scatter, and by mass. The detailed results are given in Appendix C. All dust concentrations by number are expressed as particles per cubic centimetre (p/cm^3). The mean number is introduced here as the geometric mean of the concentrations assessed by the three types of instruments - thermal precipitators, impingers, and konimeters.

Light-scatter is measured with the tyndalloscope and is given in the relative unit - $\sin^2\theta$ where θ is the angle between the polarizing screens. All mass concentrations are expressed in milligrams per cubic metre (mg/m^3).

Many references are made in this chapter to comparisons between estimates of dust concentration by various physical parameters of the dust particles as well as by various types of instrument. The ratios of the two dust concentrations being compared are termed 'comparison indices'. When comparing two physical parameters, these will be abbreviated to 'mass/number' index, 'mass/light scatter index', or 'number/light scatter' index. These are given as straight ratios of the measured dust concentrations or 1000 times greater or less than these, whichever gives a convenient range of numbers. The mass/number index follows European usage where it is defined as $(mg/m^3)/(10^{-3}.p/cm^3)$.

To assist in understanding the analysis and presentation of results, the comparison between respirable mass, as estimated by the Hexhlett, and number count, as estimated by the Gathercole konimeter, is shown in Figure 4-1. In this, the concentrations estimated by each instrument in each dust cloud are shown plotted against each other. The very wide spread can be seen; at a number concentration of about 1200 p/cm^3, the mass concentration varies from 0.6 to 5.4 mg/m^3 or over a range of 9 to 1. Also shown are the 'best fit' relationships for selected types of dust cloud.

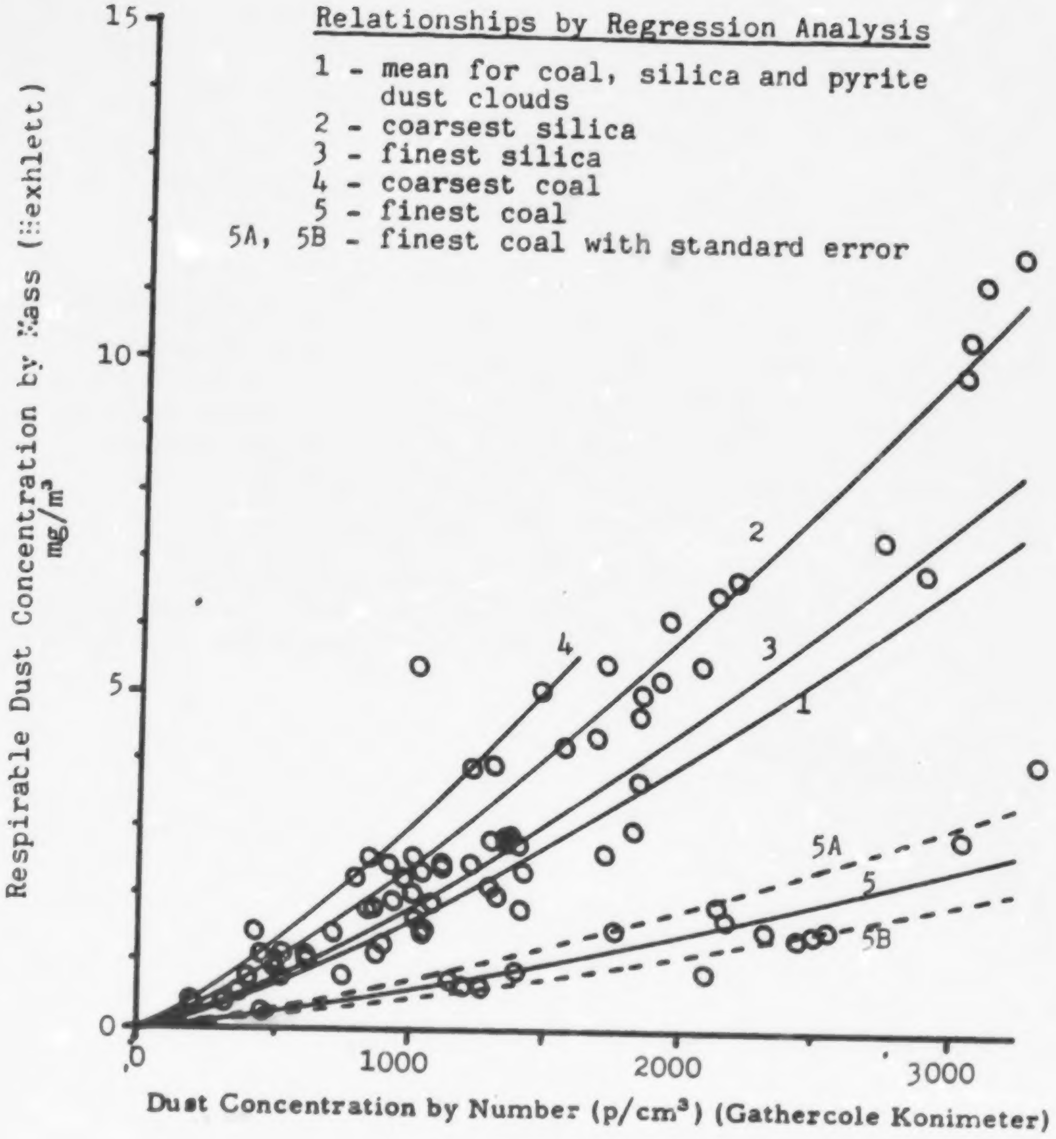


FIGURE 4. 1: COMPARISON OF DUST ASSESSMENTS BY MASS AND BY NUMBER.

TABLE 4-1

Accuracy of the Dust Estimates by Each Instrument

Group	Ratio of Individual Instrument to Group Mean	Geometric Standard Error for Single Dust Estimate
Respirable Mass		1.16
Hexhlett	0.98	
NCB MRE	1.02	
Mean Number Range		1.19 to 1.32+
Light Scatter		1.15*
Tyndalloscope		
Total Mass		1.20
Electrostatic	0.97	
104 ℓ/min Open Filter	1.02	
23 ℓ/min Open Filter	1.01	

+ Detailed results for the instruments assessing dust by number were given in Chapter 3.

* This estimate is based on the mean of twenty readings and the variability of the calibration.

ACCURACY OF MEASUREMENT BY EACH INSTRUMENT TYPE

Table 4-1 shows the mean differences between the instruments of each type and the standard error of a single estimate of the dust concentration. Also given in this table are the results obtained for gravimetric samplers measuring the total dust, i. e. that including the coarse non-respirable particles.

The standard error for an estimate of respirable mass is large because of errors due to the low weights collected on the 2. 5 ℓ/min samples.

THE COMPARISON INDICES

The mean ratios obtained between estimates of respirable dust by mass, number and light-scatter are shown in Table 4-2. Also given is the ratio of total to respirable mass. It can be seen that the range of each index is very large: 4. 8 to 1 for mass/number, 13. 4 to 1 for mass/light-scatter and 16. 5 to 1 for number/light-scatter. This is much greater than the standard error of the estimate of the comparison index in any one dust cloud - 5 to 25%.

Tables 4-3 and 4-4 show, for reference, the comparisons of mass and light-scatter with the separate types of instrument assessing dust by number.

THE EFFECT OF CONCENTRATION

The main experiments were done over a concentration range of between 4 and 10 to 1 in each type of dust cloud and tests for non-linearity, i. e., variation in the comparison index with concentration, were made using the techniques described in Appendix B. It was found that the comparisons between total mass, respirable mass, thermal precipitators, impingers, and light-scatter ($\sin^2\theta$) on the tyndalloscope were linear within the limits of statistical error, though the comparisons between these instruments and the konimeters showed significant non-linearity as discussed in Chapter 3.

In the further comparisons at lower concentrations, linearity was maintained between respirable mass, total mass, and number, by impingers and thermal precipitators. The light-scatter instrument was too insensitive to measure these low dust concentrations.

THE EFFECT OF PARTICLE SIZE DISTRIBUTION

Data on size classifications of these dusts, in terms of falling speed (comparison of total and respirable mass), are given in Table 4-2 and, in terms of projected area on thermal precipitator slides, are given in Appendix A. Correlation analysis showed that there is a statistically significant effect of dust size classification on the instrument comparison index between most pairs of instruments. Figures 4-2, 4-3, and 4-4 show plots of the comparison indices against three size classifications for the dust clouds prepared from coal, silica, pyrite, and mica. These show a marked increase in mass/number and mass/light-scatter indices with increasing coarseness of the dust cloud, while the number/light-scatter index shows no definite trend.

THE EFFECT OF DENSITY

Figures 4-2, 4-3, and 4-4 show the effects of size classification on the mass/mean-number, mass/light-scatter, and mean-number/light-scatter comparison indices respectively. It can be seen that there are marked differences between the plots for different minerals in some of the figures but not in others.

However, only for the two top figures in Figure 4-2 is there a definite grading with density thus confirming the expected effect of density on the mass/number index. Figures 4-3 and 4-4 suggest that there may be an effect due to opacity rather than density on the mass/light-scatter and number/light-scatter indices as the minerals if anything, split into the two groups, coal-pyrite and silica-mica.

THE EFFECT OF PARTICLE SHAPE

Silica, mica, glass fibre, and asbestos were chosen to give clouds with near-cubical, plate-like, fibre, and fine-fibre particle shapes respectively. The plots against size classification in Figures 4-2 to 4-4 suggest that the effects of the differences between silica and mica are small. The fibrous dust clouds, particularly those of glass fibre, tend to give extreme values of the comparison indices (Tables 4-2, 4-3, and 4-4). It is difficult, however, to separate shape effects from those due to the great differences in size distribution. The fibrous dusts consist of three components: fine particles, fine fibres, and coarse matts of fibre with very few particles in the 2 to 10-μm size range, whereas the other clouds have a smooth distribution from 0.5 to 10 μm.

TABLE 4-2

Instrument Comparison Indices between Mass Number and Light Scatter

	$\frac{\text{Respirable Mass}}{\text{Mean Number}}$ $\frac{(\text{mg/m}^3)\cdot 10^{3*}}{(\text{p/cm}^3)}$	$\frac{\text{Respirable Mass}}{\text{Light Scatter}}$ $\frac{(\text{mg/m}^3)}{(\sin^2\theta)}$	$\frac{\text{Mean Number}}{\text{Light Scatter}}$ $\frac{(\text{p/cm}^3)\cdot 10^{-3}}{(\sin^2\theta)}$	$\frac{\text{Respirable Mass}}{\text{Total Mass}}$
Coal				
Aggregated				
Full Strength	4.6	591	128	0.61
Dilute	3.8	296	76	0.70
Jet Milled	1.8	95	57	0.84
Pulverised	2.0	190	80	0.64
Cyclone	5.0	420	80	0.61
No Cyclone	5.3	390	69	0.39
Silica				
Jet Milled	3.15	95	31	0.74
Pulverised	2.9	270	91	0.47
Cyclone	3.5	170	48	0.57
No Cyclone	3.8	150	40	0.37
Pyrite				
Jet Milled	1.25	150	132	0.80
Pulverised	2.5	135	56	0.60
Cyclone	4.05	330	79	0.44
No Cyclone	3.9	350	87	0.24
Mica				
Jet Milled	3.8	125	35	0.84
Pulverised	2.55	130	49	0.83
Cyclone	3.3	140	44	0.71
No Cyclone	3.85	160	44	0.71
Glass Fibre				
Jet Milled	6.0	51	9	0.28
Pulverised	6.0	44	8	0.60
Asbestos				
Jet Milled	1.8	75	43	0.64
Pulverised	2.1	120	60	0.74

* referred to as the Mass Number Index in U.K. Publications.

TABLE 4-3

Instrument Comparison Indices: Mass/Number

Material and Dispersion Method	Mass Number Index $(mg/m^3) . 10^3/(p/cm^3)$				
	Total Mass	Hexhlett	Hexhlett	Hexhlett	Hexhlett
	TP* 1-5 μm	TP* 1-5 μm	TP* 1.2-5 μm	Impinger	Konimeter
Coal					
Aggregated					
Full Strength	16.7	9.7	6.0	4.8	3.3
Dilute	10.3	7.3	3.6	6.1	2.6
Jet Milled	3.8	3.5	1.1	6.5	0.74
Pulverised	6.4	3.7	1.35	8.3	0.74
Cyclone	12.0	7.2	3.9	12.3	2.7
No Cyclone	18.8	7.2	3.8	12.0	3.2
Silica					
Jet Milled	9.3	6.3	1.95	8.9	1.8
Pulverised	13.2	5.5	1.9	7.3	1.8
Cyclone	10.9	5.9	2.75	7.4	2.15
No Cyclone	18.5	6.7	3.0	8.45	2.1
Pyrite					
Jet Milled	7.0	5.5	0.68	3.3	0.87
Pulverised	12.4	6.95	1.0	7.1	2.2
Cyclone	17.1	7.6	3.6	7.6	2.3
No Cyclone	32.2	7.8	3.7	7.2	2.2
Mica					
Jet Milled	9.4	7.65	2.6	8.4	2.5
Pulverised	10.0	7.5	1.75	8.2	1.6
Cyclone	13.1	8.35	2.6	6.7	2.15
No Cyclone	12.9	9.6	2.9	7.9	2.5
Glass Fibre					
Jet Milled			7.2	19	1.25
Pulverised			4.8	34	1.3
Asbestos					
Jet Milled			1.35	6.9	0.66
Pulverised			1.75	5.6	0.96
Geom.+ Stan. Error Max.	1.12	1.12	1.14	1.18	1.26
Min.	1.11	1.11	1.10	1.12	1.20

* Thermal precipitator with size range.

+ In dust clouds prepared by dry dispersion from coal, silica, pyrite and mica.

TABLE 4.4

Instrument Comparison Indices: Number/Light Scatter

Material and Dispersion Method		Instrument Comparison Index $(p/cm^3) . 10 /(\sin^2\theta)$			
		TP* 1-5 μm Tyndalloscope	TP* 1/2-5 μm Tyndalloscope	Impinger Tyndalloscope	Konimeter Tyndalloscope
Coal					
Aggregated					
Full Strength		63	104	113	179
Dilute		42	83	47.5	111
Jet Milled		26	88	15.5	135
Pulverised		49	140	23	159
Cyclone		59	110	31	149
No Cyclone		53	99	30	110
Silica					
Jet Milled		14	49	11.5	54
Pulverised		46	145	35	147
Cyclone		28	63	23	76
No Cyclone		21	50	18	69
Pyrite					
Jet Milled		26	230	49	206
Pulverised		19	135	20	66
Cyclone		43	92	40	135
No Cyclone		45	96	46	152
Mica					
Jet Milled		15.5	49	16	57
Pulverised		17	75	17	90
Cyclone		16	55	22	69
No Cyclone		16	55	21	75
Glass Fibre					
Jet Milled			7.6	3	32
Pulverised			10	1.5	36
Asbestos					
Jet Milled			55	12	117
Pulverised			70	22	137
Geom.+ Stan. Error	Max.	1.16	1.19	1.25	1.26
	Min.	1.15	1.15	1.17	1.17

* Thermal precipitator with size range.

+ For dust clouds prepared by dry dispersion from coal, silica, pyrite and mica.

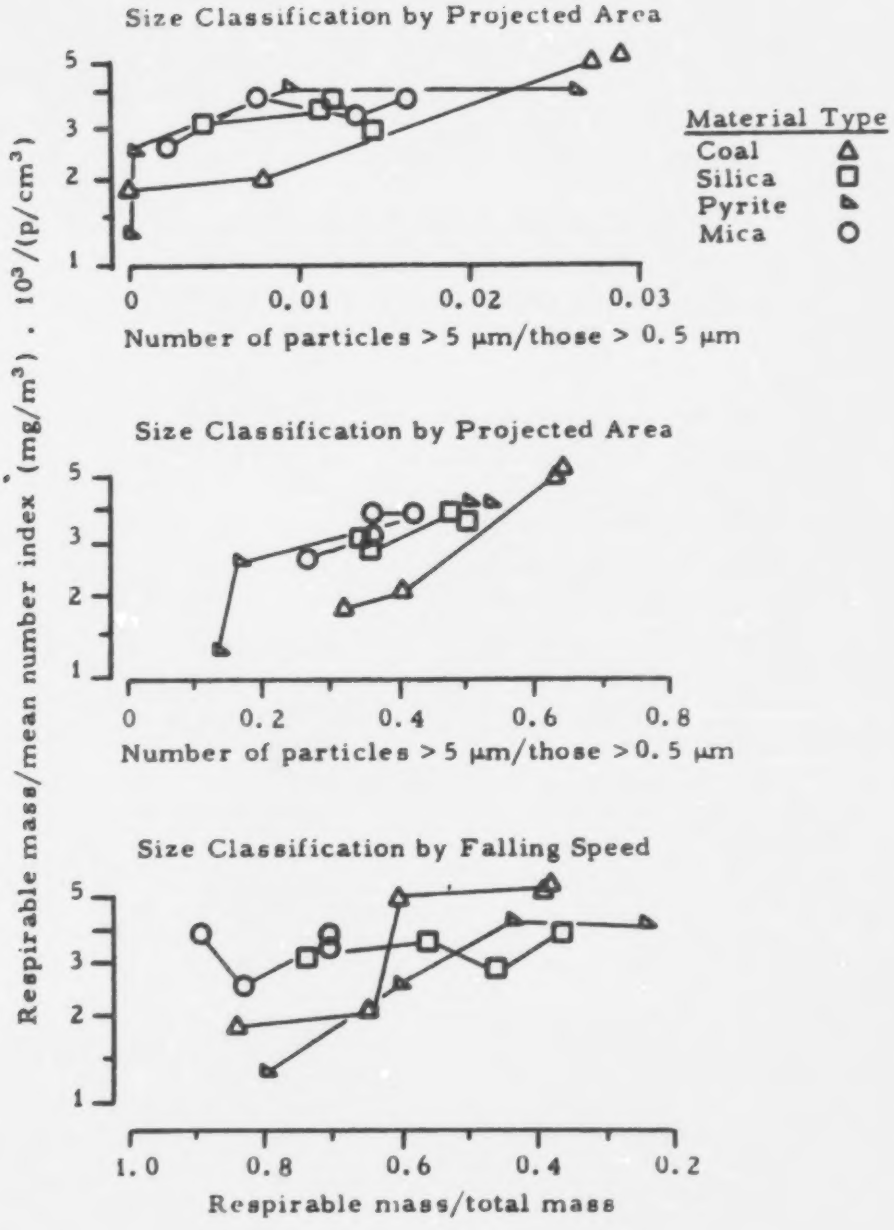


FIGURE 4-2: Effect of size classification, density and shape on respirable mass/mean number comparison index.

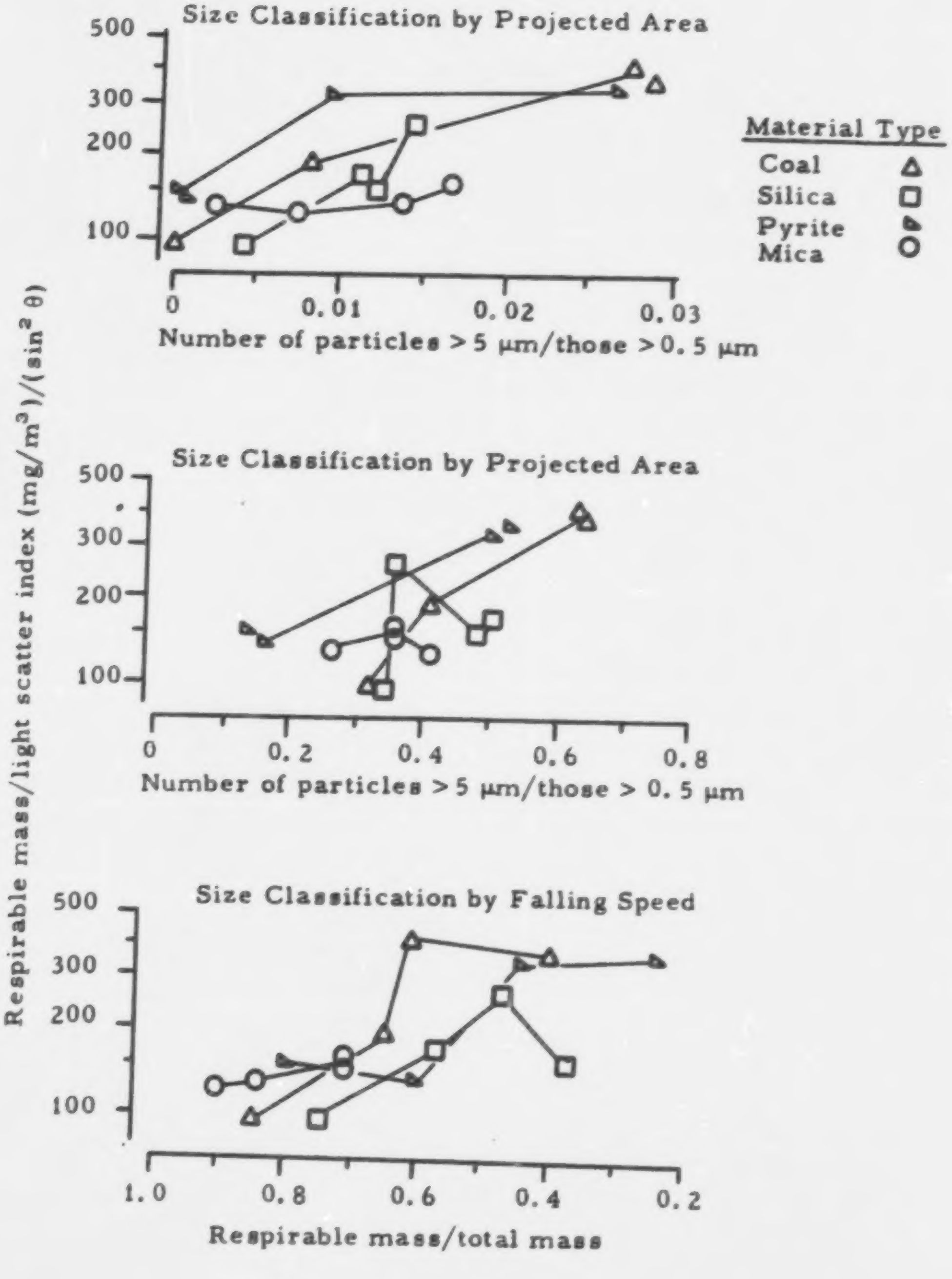
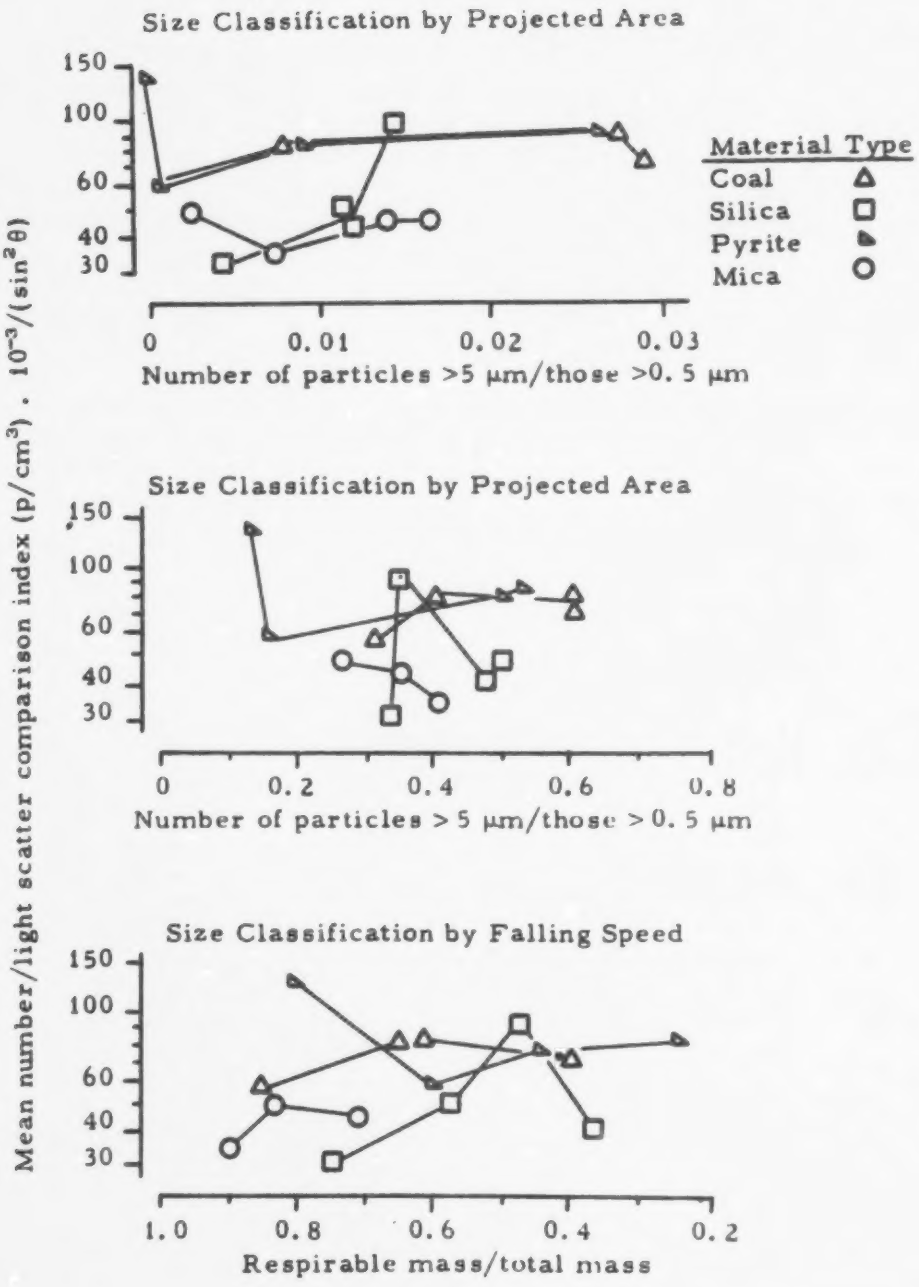


FIGURE 4-3: Effect of size classification, density and shape on respirable mass/light scatter comparison index.



Size Classification by Projected Area

Size Classification by Projected Area

Size Classification by Falling Speed

FIGURE 4-4: Effect of size classification, density and shape on mean number/light scatter comparison.

THE EFFECT OF AGGREGATION

Tables 4-2, 4-3, and 4-4 show that the comparison indices in the aggregated coal dust clouds differ considerably from those in the non-aggregated ones and tend to extreme values when compared to all the other mineral dusts. This shows that aggregation has a great effect on instrumental assessments of dust and could be important in some mining situations.

CHAPTER 5
COMPARISONS OF 'RESPIRABLE DUST' SIZE SELECTORS

A number of tests have been carried out in the laboratory dust chamber in which the 'respirable dust' concentrations, estimated by a number of dust samplers fitted with horizontal-elutriator or cyclone size selectors, have been compared. The comparisons were intended to determine the variability of each type of size selector and to compare the various types available.

Most of the comparisons were carried out in two types of dust clouds, fine coal (jet milled) and coarse coal (pulverized, no cyclone), chosen to represent the extremes of size distribution with near-cubic particles.

All dust concentrations are given in milligrams per cubic metre (mg/m^3) or relatively to that given by one chosen instrument, usually the Hexhlett.

THE EFFECT OF DESIGN DETAIL ON THE PERFORMANCE OF THE HORIZONTAL ELUTRIATOR SIZE SELECTOR

In the course of some experiments in this laboratory, it became apparent that there were some minor differences in the performance of various respirable dust samplers fitted with horizontal elutriators. These were carefully compared at the MRC design flow rate (1) in two coal dust clouds, one fine (Dispersion Method 1) and one coarse (Dispersion Method 4).

The samplers differed in the filter holders, some having a constriction between the elutriator channels and the filter. Diagrams of the significant construction features of each elutriator and the relative respirable concentration collected are shown in Figure 5-1.

Good agreement is obtained between the respirable dust concentrations indicated by the two laboratory samplers (top and fourth) and by the modified* Hexhlett (eighth).

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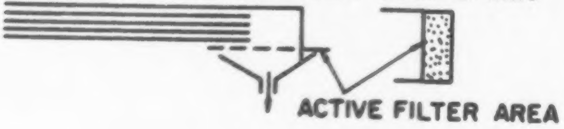
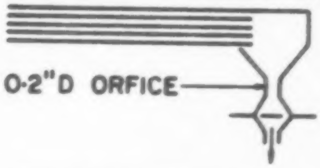
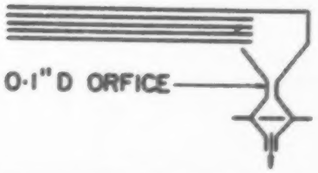
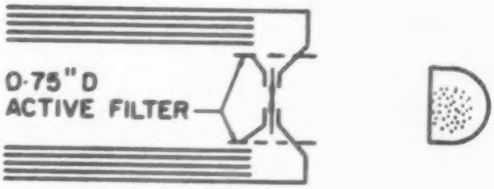
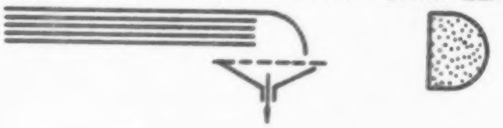
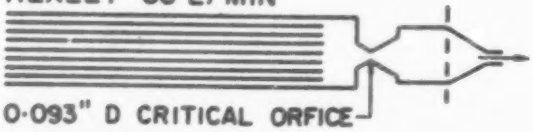
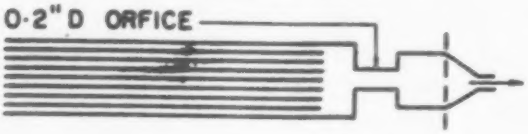
* The Hexhlett was modified by removing the flow control orifice and using the previously described (page 2-8) control valve, critical orifice and vacuum gauge.

	RELATIVE PERFORMANCE			
	FINE DUST CLOUD		COARSE DUST CLOUD	
	MEAN	SD OF MEAN	MEAN	SD OF MEAN
STANDARD LAB SAMPLER 2·83 L/MIN  ACTIVE FILTER AREA	1.0		1.0	
EFFECT OF FILTER HOLDER 	0.93	± .03	0.87	± .04
	0.86	± .03	0.83	± .04
 0·75"D ACTIVE FILTER	1.008	± .03	0.97	± .04
	0.94	± .03	0.97	± .03
NCB MRE GRAVIMETRIC SAMPLER 	0.965	± .06	1.015	± .025
HEXLET 50 L/MIN  0·093" D CRITICAL ORFICE	0.945	± .02	0.94	± .05
	1.00	± .035	1.0	± .04

FIGURES 5-1: SCHEMATIC DIAGRAMS AND RELATIVE PERFORMANCE OF RESPIRABLE-DUST SAMPLES

The Casella NCB MRE gravimetric sampler (sixth) is in fair agreement with those mentioned above. However, the flow rate is slightly higher at 2. 55 ℓ/min than the design flow rate (2. 5 ℓ/min), and the results suggest that the turned-up ends of the plates collect a part of the airborne dust leaving the elutriator. However, for practical application, the error is within a reasonable tolerance.

The Hexhlett (seventh), supplied with the flow-control orifice between the elutriator and the filter, indicates a respirable dust concentration that is about 5% low. This is apparently due to dust precipitation near the flow restriction and has been found on the laboratory samplers (second and third) which also have orifices. These orifices were introduced to smooth the dust distribution over the surface of the filter for X-ray assessment of quartz, unfortunately an invalid approach.

VARIATIONS IN PERFORMANCE OF CYCLONE SIZE SELECTORS

During the course of these studies three types of cyclones have been examined:

1. the Dorr Oliver 10-mm nylon hydroclone of which eight samples have been included;
2. the Casella chrome-plated brass cyclone (personal gravimetric sampler, type no. T13040) of which ten samples have been included;
3. the 0. 5-inch stainless steel (Unico) of which two samples have been included.

Three of the nylon cyclones were fitted, in this laboratory, with 1-inch-diameter aluminum filter holders by pushing them over a rubber seal on the extension of the vortex finder, and three were similarly fitted with plastic filter holders. The other two units were assembled by M. S. A. and Unico respectively.

Some variations, between the samplers of each type, were found and are given in Table 5. 1.

It can be seen that there are small differences between the six nylon cyclones A to F using a smooth airflow with B and C significantly lower than E and F. These differences are probably due to small differences in the assembly of the cyclones and filters.

TABLE 5. 1

Cyclone Size Selectors: Variations in Dust Collection

		Flow	Coarse Coal Dust				Fine Coal Dust			
	Pump	ℓ/	Set 1		Set 2		Set 1		Set 2	
Cyclone	type	min	RDC[1]	SD[2]	RDC	SD	RDC	SD	RDC	SD
10mm nylon										
A	CO[3]	1.7	.78	.07			.79	.08		
B	CO	1.7	.735	.06			.8	.07		
C	CO	1.7	.755	.04	.84	.05	.845	.05	.9	.06
D	CO	1.7	.77	.06	.89	.035	.84	.05	.91	.035
E	CO	1.7	.84	.08	.76	.06	.84	.07	.85	.04
F	CO	1.7	.815	.09			.83	.06		
Statistical significance of difference between extreme and mean(A-F)			0. 1		0. 05		--		0. 1	
F	PS[4]	1.7	.74	.05						
Unico	P[5]	1.7	.63	.05						
MSA	P	1.7	.49	.05						
MSA	PS	1.7	.67	.05						
Casella										
1	PS	2.0	.92	.02			.95	.04		
2	PS	2.0	.76	.04			.88	.04		
3	PS	2.0	.85	.06			.94	.05		
4	PS	2.0	.93	.06			1.02	.07		
5	PS	2.0	1.02	.06			1.03	.05		
6	PS	2.0	.96	.06						
7	PS	2.0	1.06	.06						
8	PS	2.0	1.06	.06						
9	PS	2.0	1.03	.06						
10	PS	2.0	1.03	.06						
statistical significance of difference between extreme and mean			0. 02				0. 1			
1/2 " steel										
1	CO	8.15	1.25	.09			.935	.05		
2	CO	8.15	1.23	.07			.90	.05		
1	CO	16.15	1.0	.06			.84	.02		
2	CO	16.15	1.07	.12			.80	.05		

Footnotes:

1 RDC - Dust concentration relative to the Hexhlett.
2 SD - Standard deviation.
3 CO - Smooth flow controlled by critical orifice.
4 PS - Diaphragm pump with pulsation damper.
5 P - Diaphragm pump, no damper.

It can be seen that the M.S.A. and Unico samplers used did not give the same size selection as the 10-mm nylon cyclone used with a smooth flow. Of the ten Casella cyclones, only one (No. 2) showed a statistically significant difference from the others. Examination of this cyclone showed that the entry slot was narrower than on the other cyclones, 0.058 as compared with 0.062 to 0.064 for the others. This dimension is outside the tolerance permitted.

The difference between the two 0.5-inch stainless-steel cyclones was not statistically significant; however, it should be noted that these are assembled by hand and should always be checked.

COMPARISON OF CYCLONE AND HORIZONTAL-ELUTRIATOR SIZE SELECTORS

A number of tests comparing cyclone and horizontal-elutriator size selectors have been done. The first set of comparisons was made between four 10-mm nylon cyclone and four horizontal-elutriator size-selective samplers. For these tests, the cyclone size-selective samplers were made up from the nylon cyclone and a 1-inch diameter filter holder mounted directly over the outlet end of the vortex finder and operated at 1.3, 1.65, 1.95, and 2.6 ℓ/min respectively, using constant flow, controlled by critical orifices. The horizontal-elutriator size-selective samplers consisted of a 2.5-ℓ/min Casella, a 2.83-ℓ/min laboratory model, a 50-ℓ/min standard Hexhlett, and a Hexhlett run at 25 ℓ/min. The first three were intended to operate at the standard Medical Research Council (1) top cuts of 7.1 μm for unit density spheres, and the fourth had a theoretical top cut of 5.0 μm.

The comparisons were carried out in thirty dust clouds of thirteen types chosen to cover the range of the standard laboratory types. The results are given in detail in Appendix D and are summarized in Figures 5.2 and 5.3. Figure 5.2 shows a plot of the ratios of dust concentrations - estimated by samplers fitted with cyclone size selectors (operated at a range of flow rates) - to those estimated by samplers fitted with horizontal-elutriator size selectors. This graph shows both the mean and the standard deviation of all the measurements at each flow rate. Figure 5.3 shows the relationship between the ratio and a measure of the dust particle size in the dust clouds.

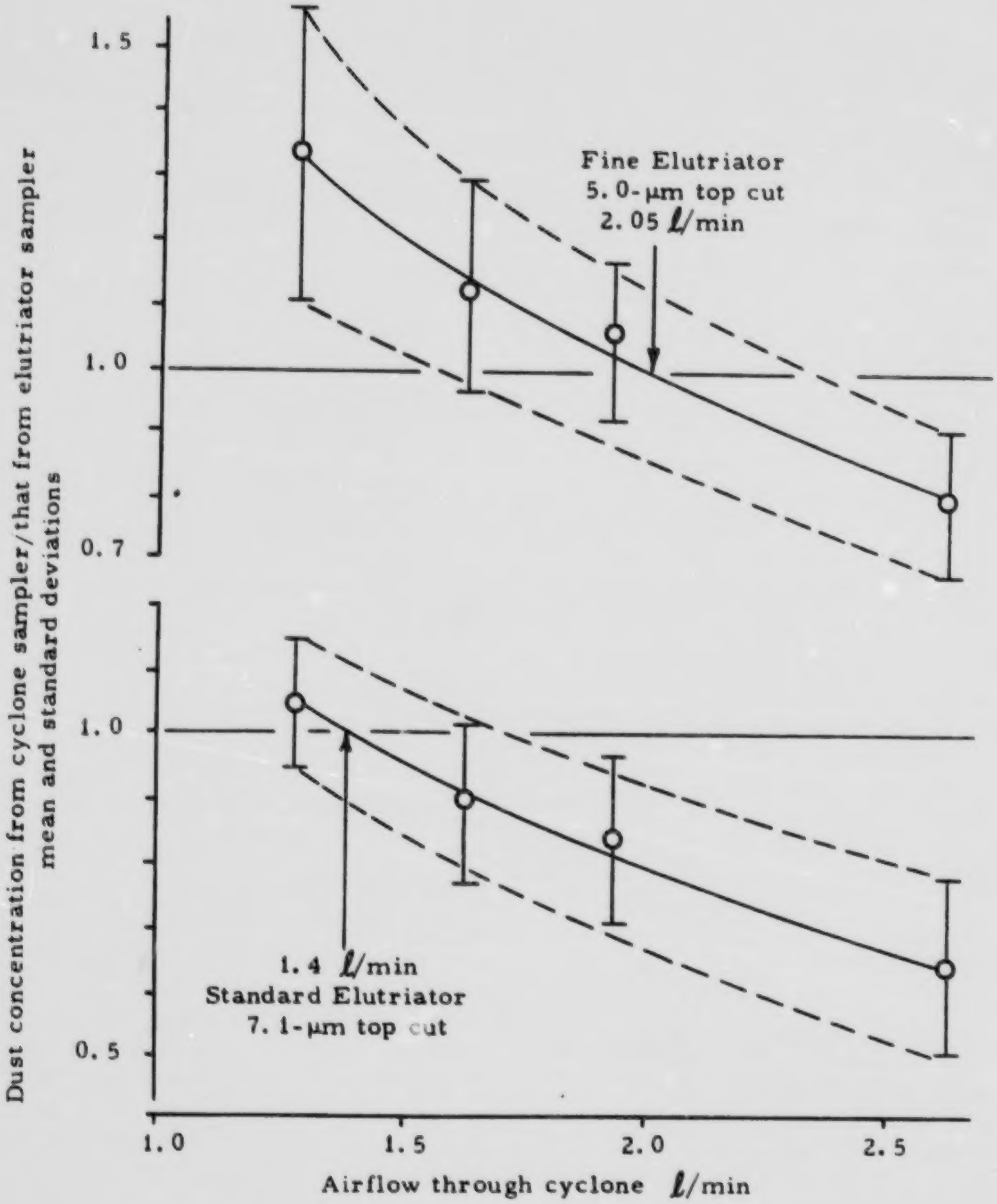


FIGURE 5-2: Effect of airflow on respirable dust concentration estimated by samplers with cyclone size selector.

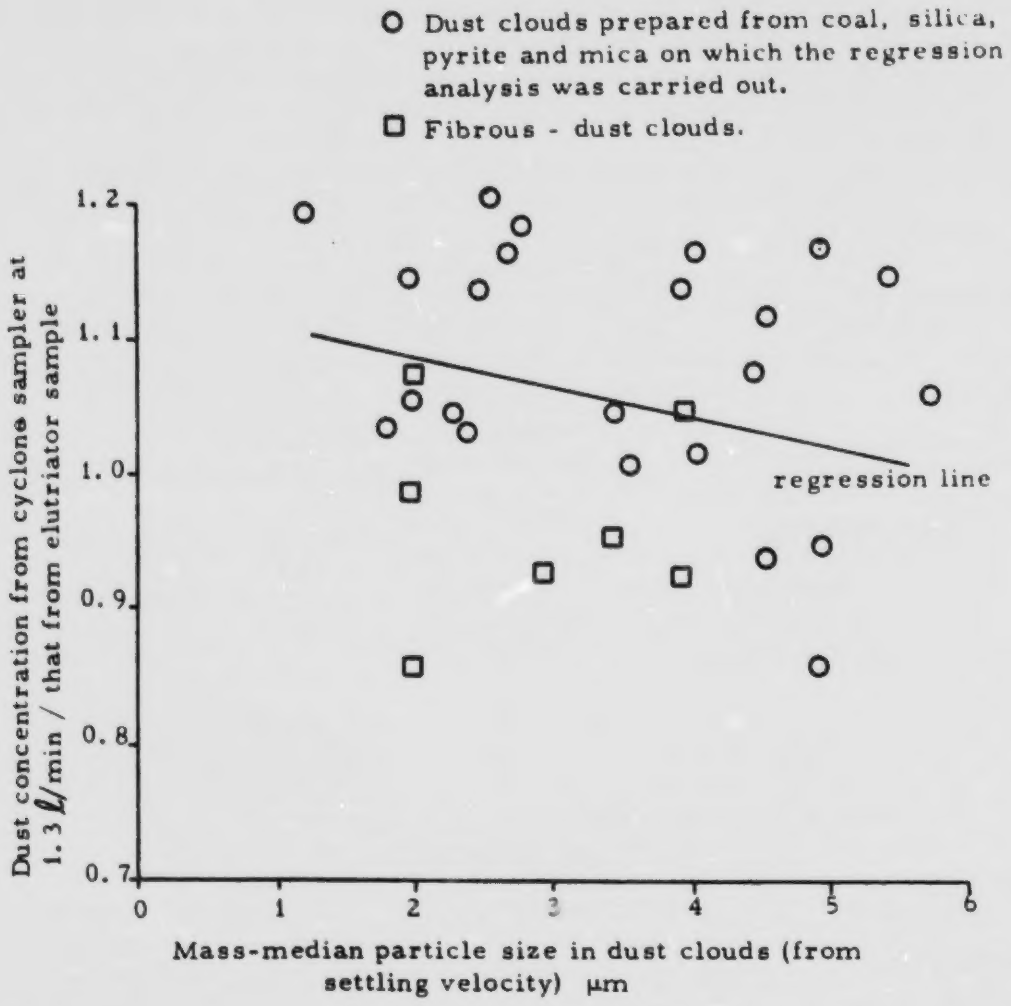


FIGURE 5-3: The effect of dust size distribution on comparison between cyclone and horizontal-elutriator size selectors.

In the second set of comparisons, three types of cyclones, recommended for size selective sampling, were compared with the modified Hexhlett (critical orifice placed after the filter) in the fine and coarse types of coal dust cloud. The results are given in Appendix D and are shown in Figure 5.4 as plots of the relationship between dust concentration relative to the Hexhlett and the airflow through the cyclone. Log-log scales are used so that the curves have the same appearance for each cyclone.

It is apparent that there are differences in behaviour between the three types of cyclone with changes in airflow and dust size distribution.

The cyclones differ in material, which could affect the conductivity and collection characteristics for charged particles, and in aspect ratio of the entrance slit; the nylon has a square slit (0.080 x 0.080 inch), the Casella has a long slit (0.060 x 0.375 inch), and the 1/2-inch cyclone has a double entry long slit (0.15 x 0.75 inch).

For the 10-mm nylon cyclone, best agreement with a sampler fitted with a horizontal elutriator (Medical Research Council specification) is found at a flow rate of 1.0 to 1.2 /min in the second set of comparisons, whereas in the first it was at 1.4 /min. The differences are due to changes in experimental technique, permitting the collection of larger samples, to the more accurate operation of the horizontal elutriator samples, and to elimination of some errors as discussed in the first section of this chapter.

The Casella cyclones give best agreement at a flow rate of 2.1 /min which is in fair agreement with Higgins and Dewells calibration of 1.9 /min to agree with the Hexhlett, which presumably reads low because of its critical orifice.

The 0.5-inch stainless-steel cyclones show very different calibrations, 6 and 15 /min, in the fine and the coarse coal dust clouds respectively. The results obtained suggest that this type of cyclone has a size collection characteristic that is markedly different than those of the other cyclones and that, in routine sampling, appreciable differences could be found if this and other types of cyclone are used together.

The advantages and disadvantages of the various types of cyclone size selectors are laid out below.

Advantages

Nylon - most consistent within limited number of specimens,
- low price (injection moulded).

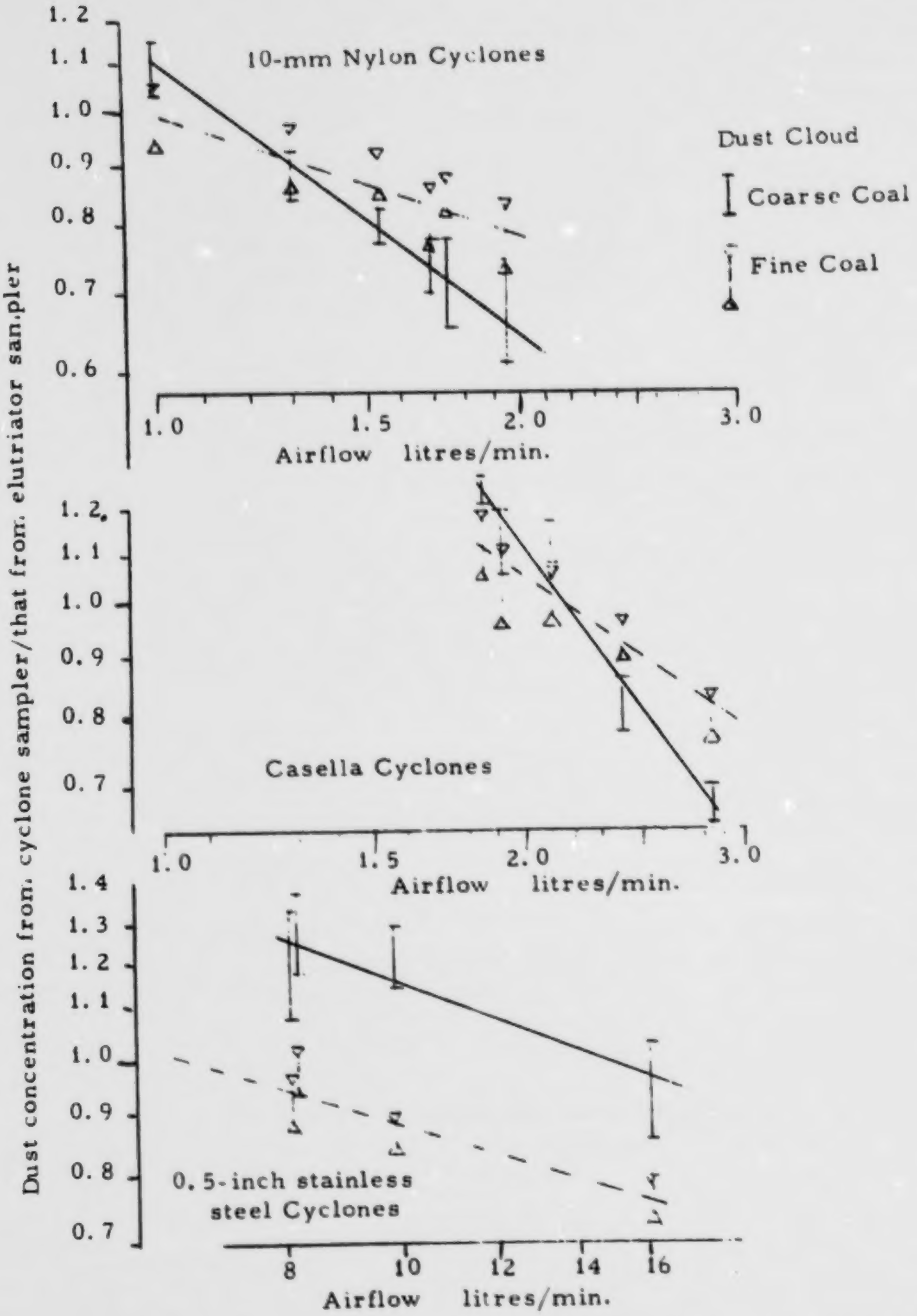


FIGURE 5.4: Effect of airflow and dust size on the 'respirable dust' concentration estimated by samplers using cyclone size selectors.

Casella - most suitable flow rate for personal sampler,
- mass collected has least dependence on flow rate,
- conductivity of metal surfaces closest to that of the wet bronchial passages.

0.5-inch - high flow rate suitable for compositional samples,
- conductivity of metal surfaces closest to that of the wet bronchial passages.

Disadvantages

Nylon - low flow rate,
- low conductivity.

Casella - inaccuracy of manufacture?,
- cost.

0.5-inch - variation with size distribution.

DISCUSSION

It has been shown in this chapter that relatively small changes in construction can affect respirable dust size selection appreciably and it is therefore recommended that all aerodynamic size selecting dust samplers should be checked against a 'standard' instrument prior to use. This can most conveniently be done in a dust chamber with a dust cloud containing about 50% respirable dust.

REFERENCES

1. Hamilton, R.J. and Walton, W.H., "The Selective Sampling of Respirable Dust", in 'Inhaled Particles and Vapours', C.N. Davies (Ed.), Pergamon Press, Oxford (1961).

A good review of the literature on size selective sampling is,
Lippmann, M., "Respirable Dust Sampling", A.I.H.A. Journal, Vol. 31, March-April (1970).

APPENDIX A
DUST CLOUD PREPARATION AND PROPERTIES

INTRODUCTION

The laboratory dust chamber was originally erected in Ottawa by L. Richards. The chamber was moved to Elliot Lake in 1965, and has since been used mainly on the program, "Comparison of Dust Sampling Instruments". The requirements of this program led to considerable extensions of some of the dust chamber ancillary apparatus. This section describes the preparation, sizing, feeding, and dispersion of material and some properties of the resulting pure mineral dust clouds such as uniformity in time and space and size distribution.

The preparation of a dust cloud can be separated into a number of stages: preparation of the material, feeding, and dispersion.

DUST SOURCES

A limited selection of minerals was chosen to cover the main range of density and particle shape found in Canadian mines. These were - for density- coal, silica, and pyrite and -for shape - silica, mica, and asbestos. Because asbestos gives very fine fibres less than 0.1 μm in diameter and below the resolving power of a microscope, a glass fibre about 0.5 μm in diameter was also examined. The resulting dust clouds are regarded as standard for laboratory purposes and stocks of material are available for future requirements.

The materials were received from the sources in the forms stated below:

1. Coal:
   Source: Princess Colliery, Nova Scotia.
   Designation: Washed nuts S13 1.75 to 0.75-inch ring size.

2. Silica:
   Source: Ottawa Silica Company, Illinois.
   Designation: ASTM C-109
   Size: 16 to 100-mesh.

3. Pyrite:
   Source: Nordic Mine, prepared by flotation at Stanleigh Research Lab.
   Purity: 95%; quartz is main impurity.

4. Mica:
   Source: A Quebec molybdenite mine, supplied through Q.M.M.A.P.A. by courtesy of Mr. Grassmuck.
   Purity: The mica contained quartz and molybdenite lumps.
   Sorting: Flakes of visibly pure mica were hand sorted from the partially crushed source material.
   Size: 1 to 2 inches.

5. Asbestos:
   Source: Californian asbestos deposits; a fine fibre received through the Department of National Health and Welfare by courtesy of Dr. Villiers. Various Canadian asbestos samples were tried out but none gave a dust cloud with as high a proportion of fibrous particles as this particular material.

6. Glass Fibre:
   Source: Gelman (filter material manufacturers), Michigan.
   Designation: Type GF/A glass fibre filter paper.

DUST PREPARATION

The equipment available in this laboratory for dust preparation is:

1. laboratory crusher, 4.5 x 3.25-inch opening, Denver Fireclay Co.;
2. pulverizer, type VD, Bico Inc.;
3. wet grinding mill, M. 18, SWECO;
4. twin jet fluid energy mill, Helme Products Inc.;
5. sieves, 8-inch, and sieve shaker.

This equipment is used as found necessary to produce the desired dust. The glass fibre filter paper was chopped into small pieces, less than 0.125 inch square.

For preparation of non-aggregated dust clouds, dry processes were used throughout, either the Bico pulverizer or the Helme mill being the final breakage process. For these, standard operating conditions were chosen; the Bico pulverizer plate gap was set at 0.005-inch gap, and the Helme jet air supply pressure was run at full bore ranging over 80-85 lb/in^2 dependent on the compressor cycle. For preparation of aggregated dusts, the Helme mill is used for the softer materials and the Sweco for the harder materials. The desired material is a fine slurry in alcohol and/or water.

DUST FEEDING

The methods used to feed dust can be divided into two categories, those for dust clouds of non-aggregated particles and those for dust clouds containing aggregates. As it is very difficult to separate slightly damp fine dust particles, all the processes used to produce non-aggregated dust clouds have used dry material. Dust clouds with controlled aggregation have been produced by spraying fine dust dispersed in a volatile liquid; the technique is described later. The feed problems with sprays are producing low flow rates of liquids containing high concentrations of suspended particles; they are part of the dispersion method and will be described under it.

The main requirement of the feed method is that it will give a uniform dust concentration in the chamber over the period of the run. Two factors which interfere with this in handling dry dusts are - variations in volume rate of material feed and variations in size distribution of the material. The feed material often contains a large proportion of dust, coarser than 10 µm, that gets deposited prior to the chamber; the important parameter of the size distribution of the feed is the ratio of fine to coarse material. The variations that arise are mainly due to either insufficient mixing or to size segregation in filling or operating the feeder.

A number of dust feeders have been examined and, though they all work reasonably well on free-flowing powders, the SMRE type feeder was found to be the most versatile.

USBM TYPE OF FEEDER

This feeder is based on the one at the United States Bureau of Mines, Pittsburgh and is illustrated in Figure A-1.

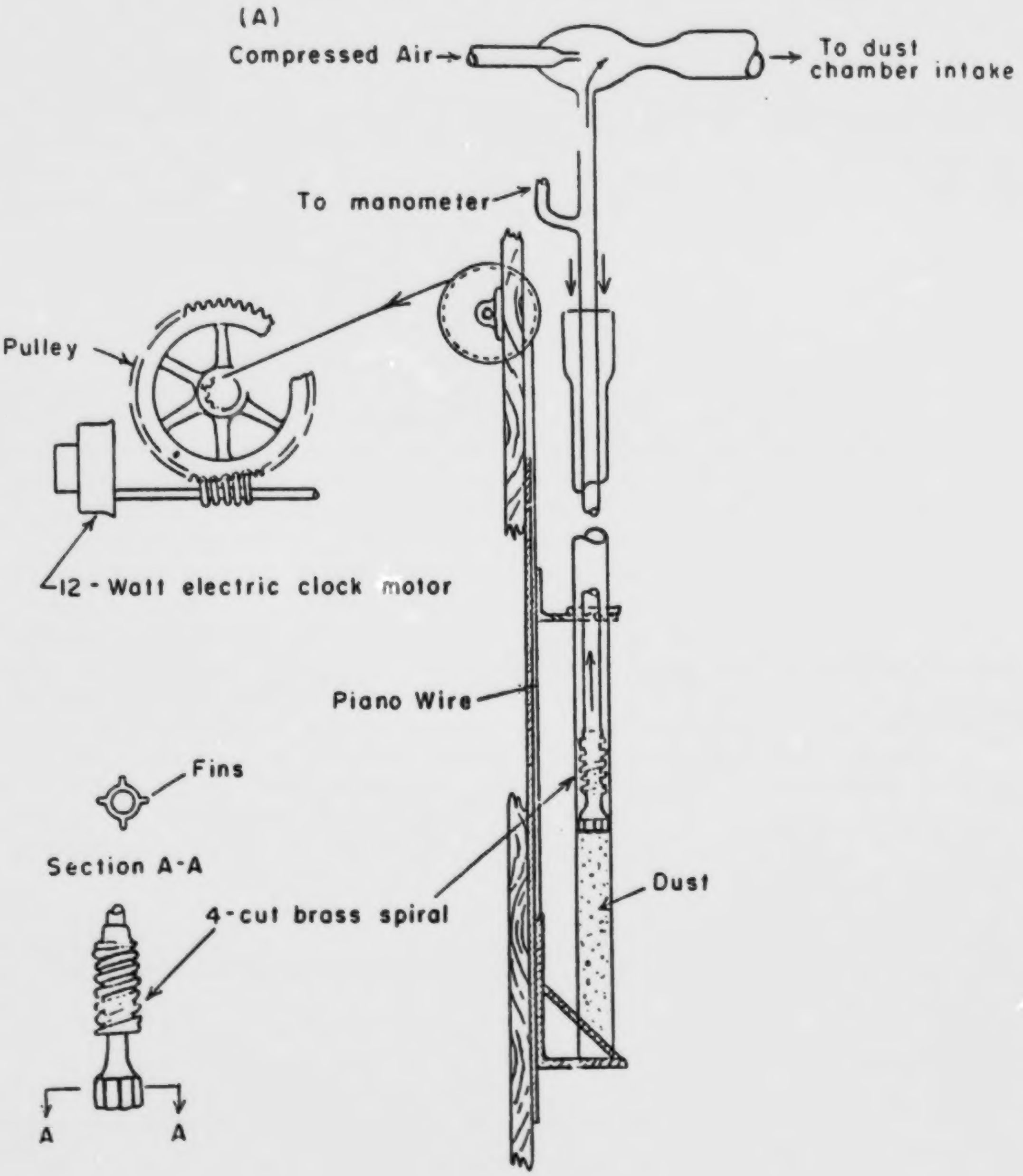

(A)
Compressed Air
To dust chamber intake
To manometer
Pulley
12 - Watt electric clock motor
Piano Wire
Fins
Section A-A
4-cut brass spiral
Dust
A
A

FIGURE A-1: DUST FEED APPARATUS

The dust is placed in a glass tube 34 inches long; the inner wall of this tube must be as smooth and true as possible. Inside the large tube fits a small glass tube which remains fixed; the outer tube traverses this tube. The inner tube is fitted with a brass spiral on the lower end which should be a slide fit to the inner wall of the larger glass tube. It is very important that the brass spiral has a snug fit so that most of the air passes through the spiral and swirls, giving a steady pick-up of the dust in the outer rising tube. The dust feed rate depends on the diameter of the outer tube and the rate at which it is elevated.

The following table gives the rise and dust rate for different-size pulleys, using a 1-rpm capacitor-start electric motor, geared to a 6-inch-diameter, 100-tooth gear.

TABLE B-1

Pulley Size and Hoisting Speed

Pulley Size in.	Rise Time in./hour	Dust Feed Rate cm^3/hour
4.5	8.62	102.15
2.5	4.75	56.75
1.5	2.87	34.05
1.0	1.94	22.7
0.75	1.44	17.
0.5	0.94	11.35

The difficulties with this feeder apparently are because of the dependence of the feed rate on the airflow through the feeder and because of the large area of dust exposed to the airstream as compared to the volume flow of dust. Though a satisfactory balance could be achieved for fine free-flowing powders, it could not be obtained for materials containing relatively coarse particles or for mica and asbestos. This feeder was used until the SMRE feeder was built.

WRIGHT FEEDER

The Wright (1) dust feeder was originally intended for coal and feeds dust by scraping a uniform thin layer off a lightly compacted dust bed. The feeder is commercially produced. Though the feeder is successful for free-flowing powder, it jams when used with fibrous materials like asbestos, and has been tried in only a few experiments.

SMRE TYPE FEEDER

Hattersley et al. (2) of the Safety in Mines Research Establishment, U.K., described a dust cloud producer. Essentially the feed portion of this consists of a grooved plate rotating slowly about a vertical axis, with a funnel and scraper blades to fill the groove and level the dust off flush with the plate surface. The dust is drawn out of the groove by an air operated ejector. The feed rate is controlled by the speed of rotation and the size of the groove. The Mines Branch version of this instrument is shown in Figure B-2 and the feed rates obtainable are shown in Table B-2.

TABLE B-2

Feed Rate of SMRE Type Dust Feeder

Groove Size		Feed Rate by Volume cm^3/hour at	
Diameter* Inch	Pitch Diameter Inch	1.08 rpm	0.054 rpm
0.062	4	20.5	1.025
0.094	5	57.5	2.88
0.125	6.375	132	6.6
0.25	4	323	16.1
0.375	6.375	1188	59.4

* Nominal diameter of groove of semi-circular cross section.

The feed rate has been reduced further by switching on and off for short periods, i.e., 5 seconds in 20.

This feeder worked well on free-flowing powders but, with coarse powders like sand or fluffy ones like mica, the scraper set across the groove can remove material from the groove at irregular intervals and hand scraping with a spatula was used. It was found possible to obtain a reasonably uniform feed of fibrous materials by hand-filling the groove.

The SMRE type feeder has proved to be the most versatile and the simplest to adjust over a wide range of dusts and feed rates. Dust clouds with concentrations in the range of 10 to 1,000,000 particles per cubic centimetre have been produced.

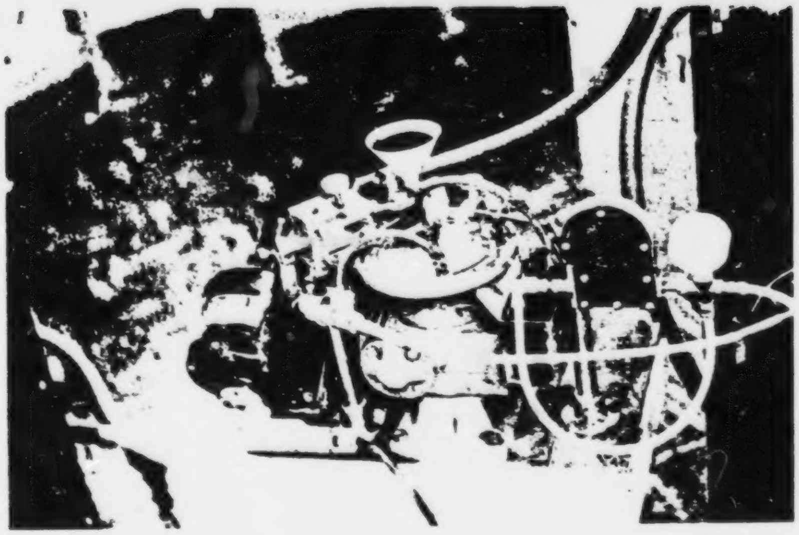


FIGURE B-2: The SMRE Type Feeder

showing:

Left:	The drive motor
Centre:	Reduction gear box with grooved plate, feed funnel, scraper blades, and suction U-tube
Right:	The Helme twin-jet fluid energy mill.

DUST DISPERSION

A number of dust dispersion methods have been used and are characterized in three ways: type of process, dry or wet; amount of fresh breakage; and intensity of air turbulence.

The first three methods are essentially for dry dusts, with the first two introducing appreciable amounts of fresh breakage on non-fibrous dusts. The intensity of air turbulence and separation of matted fibres decreases in the order given. The fourth method produces an aggregated dust cloud by atomizing a slurry of fine particles in a volatile liquid.

Jet Mill

In the jet mill (3), method 1, there are two opposed compressed air jets entering the grinding region, each drawing a dust-laden airstream, one from the feeder and the other from the cyclone chamber producing collisions between high-speed particles. The action is mainly fresh breakage of non-fibrous particles or dispersion of matted fibres.

This method produces a dust cloud having a higher proportion of fine dust than have clouds by the other methods. The jet mill is shown mounted on the right hand side of the SMRE type feeder in Figure B-2.

Pulverizer

In method 2, the material is coarser than 200 mesh; it is picked up from the feeder by a low-pressure air ejector and blown into the gap (0.005-inch) between the rotor and stator of the pulverizer. The pulverizer produces fine dust and disperses it into the air. Dust-laden air is then drawn from the pulverizer case and blown into the chamber intake airway by a second air ejector.

The main effect of the pulverizer on asbestos and glass fibre is increased dispersion of matted fibres rather than fresh breakage. For the other dusts, the fine material is broken by the pulverizer under much the same conditions as when preparing material for the third dispersion method and the main difference in the resulting dust clouds is probably due to some loss of the finest material when collecting the pulverized material prior to feeding in Method 3.

Air Ejector

In Method 3, a compressed air ejector is used to draw the dust from the feeder and blow it into the intake airstream of the chamber. The turbulence in the airstream, when supplied with air at 5 psi, is sufficient to produce non-aggregated dust clouds from coal, silica, pyrite, and mica but, even at 80 psi, it was not sufficient to separate the matted asbestos and glass fibres and gave rise to dust clouds characterized by low concentrations of mainly non-fibrous particles.

The feed material for this method of dispersion contains the fine particles which are to be dispersed in the dust cloud and all materials were prepared by pulverizing in the Bico pulverizer.

Atomization of Dust Suspension in Liquid

The object of Method 4 is to obtain dust clouds of particles with controlled aggregation. The principle is that when the liquid of a spray drop evaporates, the dust particles contained in it remain together as an aggregate. The number of particles in each drop follows a binomial distribution (4) depending on the number concentration of dust particles in the liquid and the size of the droplets. The proportion of aggregated particles increases with dust concentration and droplet size. The dust concentration in the liquid is limited by preparation and handling difficulties and the maximum drop size is limited by the problems of entrainment in the airstream and carriage into the chamber.

The experiments with compressed air atomizers were not very successful in producing dust clouds with many aggregates, because most of the droplets were too fine and gave a large proportion of single particles. Then a spinning disc generator was used. This consists of a 4-inch-diameter aluminum disc mounted on a lathe tool post grinder that turned from 7000 to 36000 rpm.

It has been found possible to obtain dust clouds in which more than 50% of the particles occur as aggregates. The optimum conditions attained have been with slurries containing 10^{10} particles, less than 5 μ in diameter, per cm^3. Dense slurries are difficult to feed slowly and steadily at a uniform rate because the jets clog frequently, therefore, the simplest technique is to spray the required amount of slurry directly into the chamber and commence sampling after the droplets have evaporated. This technique has the disadvantage that the dust cloud being sampled has a steadily decreasing concentration.

TOP SIZE CONTROL OF DUST

The dust-laden air stream from the dust dispersal mechanism can pass through an adjustable cyclone prior to joining the main intake of the chamber.

The cyclone characteristics have not as yet been ascertained because to date, only experiments, either with or without the cyclone operated at a set adjustment have been done. The effect of the cyclone was to reduce the total mass concentration of dust by 10 to 50% for coal, silica, pyrite, or mica by removing the coarser dust particles.

Even without the cyclone, very few particles larger than 10 µm are present in the chamber dust cloud. Apparently most large dust particles settle in the ducts leading to the chamber.

CHAMBER AIRFLOW

There is a forced airflow through the chamber of up to 150 cu ft/min. Intake and exhaust fans are fitted to the chamber, both have dampers to permit adjustment of airflow, through, and pressure inside, the chamber. The dust-laden air enters the chamber through a perforated duct on the floor of the chamber and is exhausted through perforated ducts up the four corners and along the top of three sides of the chamber as previously described by Richards (1). The airflow is monitored by an inclined manometer across the chamber and the inlet and exhaust ducts. Figure B-3 shows the chamber airflow plotted against this pressure drop; about 100 cu ft/min or a pressure drop of 0.4 inches W. G. is a suitable setting for most experiments. A second inclined manometer is fitted to indicate the difference in pressure inside and outside the chamber. The intake and exhaust dampers are adjusted to a slight positive 0.01-inch W. G. pressure inside the chamber with all the instruments running. A slight positive pressure is used, because, with negative pressure, slight leaks of clean air into the chamber can play on a particular instrument and cause it to give a low estimate of the dust concentration. During these experiments a high-efficiency particulate filter was added to feed clean air to the chamber and to permit working in low-concentration pure-dust clouds.

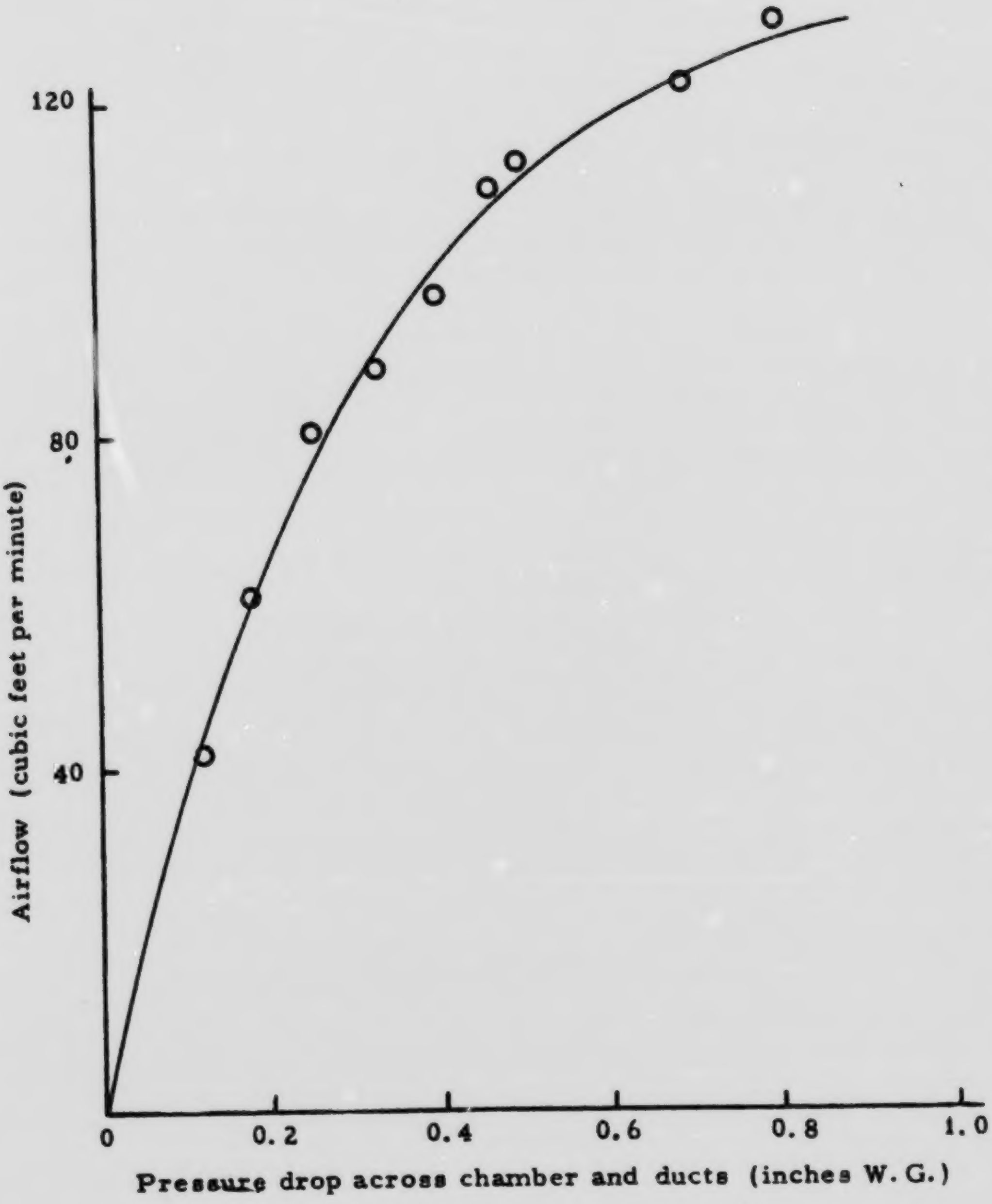


FIGURE A-3: .Airflow through and pressure drop across chamber.

A circulating fan is fitted inside the chamber. The speed, position, and direction of this was adjusted so that all instruments were exposed to air velocities of 50 to 150 ft/min. This fan left only small zones of dead air in some of the corners.

CONTROL SETTINGS FOR STANDARD DUST CLOUDS

The standard non-aggregated dust clouds are all produced using the SMRE type dust feeder and three methods of dispersion with an airflow through the chamber of 100 cubic feet per minute.

Table B-3 shows the material, method of dispersion, size of feed material, feed rate and resulting dust concentration, by number of 1 to 5-μm particles (thermal precipitators) and by mass of respirable dust (Hexhlett). Only the values obtained at the middle feed rate are given here because dust concentrations are proportional to feed rate and can hence be readily determined for other feed rates.

SOME PROPERTIES OF THE DUST CLOUDS

Variations of Concentration within the Chamber

Two dust sampling instruments, a midget impinger and an open filter were placed in various parts of the chamber, and the resulting dust concentrations were compared with those obtained by similar instruments in a set position. No significant differences in concentration were found in any of these tests. There was a slight tendency for total mass concentration to rise, by less than 5%, near the chamber floor, below the position of the lowest set instrument. This is presumably due to partial settlement of coarse non-respirable dust.

Variations of Dust Concentration with Time

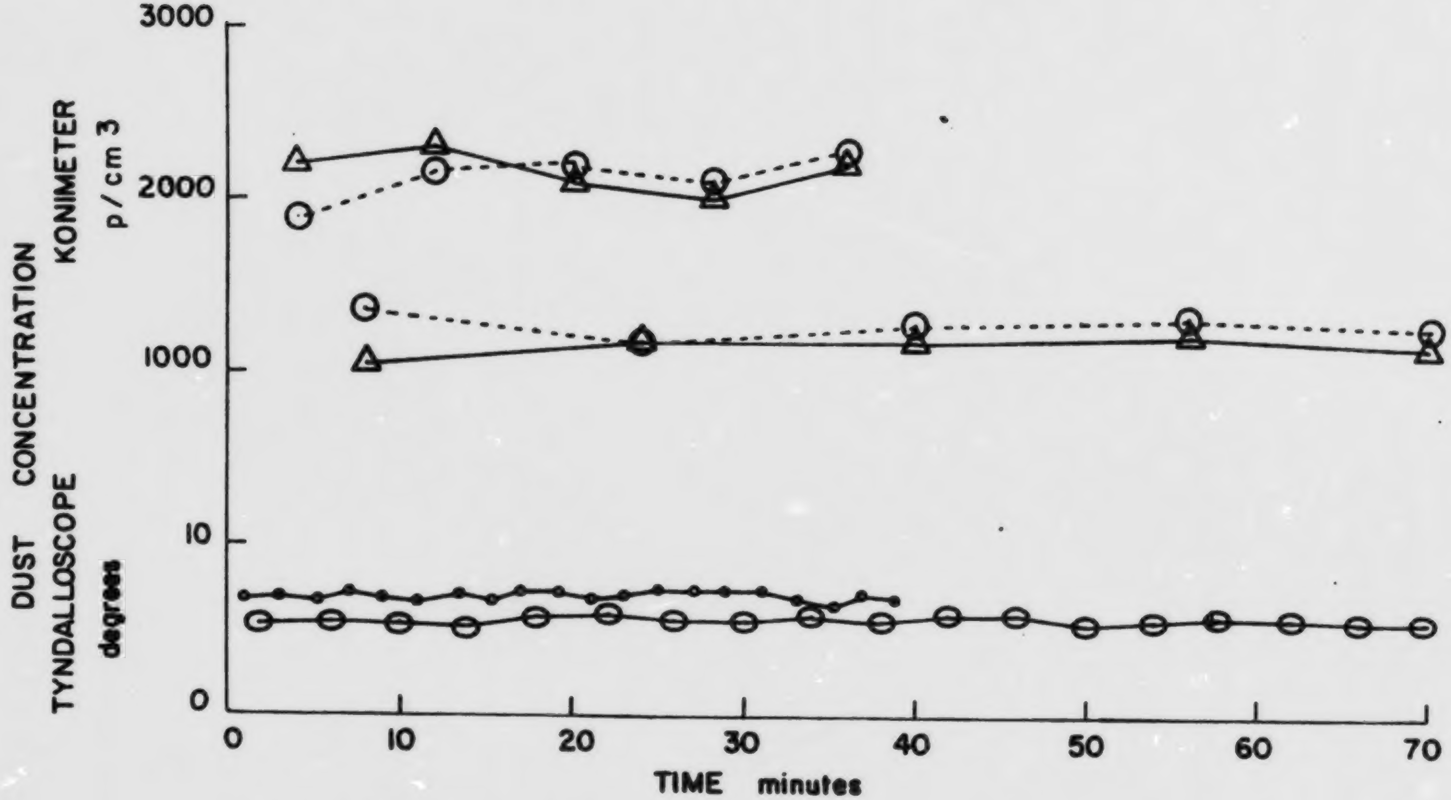
The variations with time can be separated into those within runs and those between runs with the same dust and feed settings. Figures B4 to B7 show the dust concentrations measured with the snap sampling instruments during a few typical runs. It can be seen that the variation in concentration is greater with asbestos than with the other minerals. This is due to both greater variations in feed rate because of difficulties of filling the groove evenly with asbestos and to the poor performance of the konimeter in sampling fibrous dusts. With the other minerals, the expected variation in sampling one dust cloud at one time with these instruments is almost as

TABLE A-3

Conditions for Producing Standard Dust Clouds

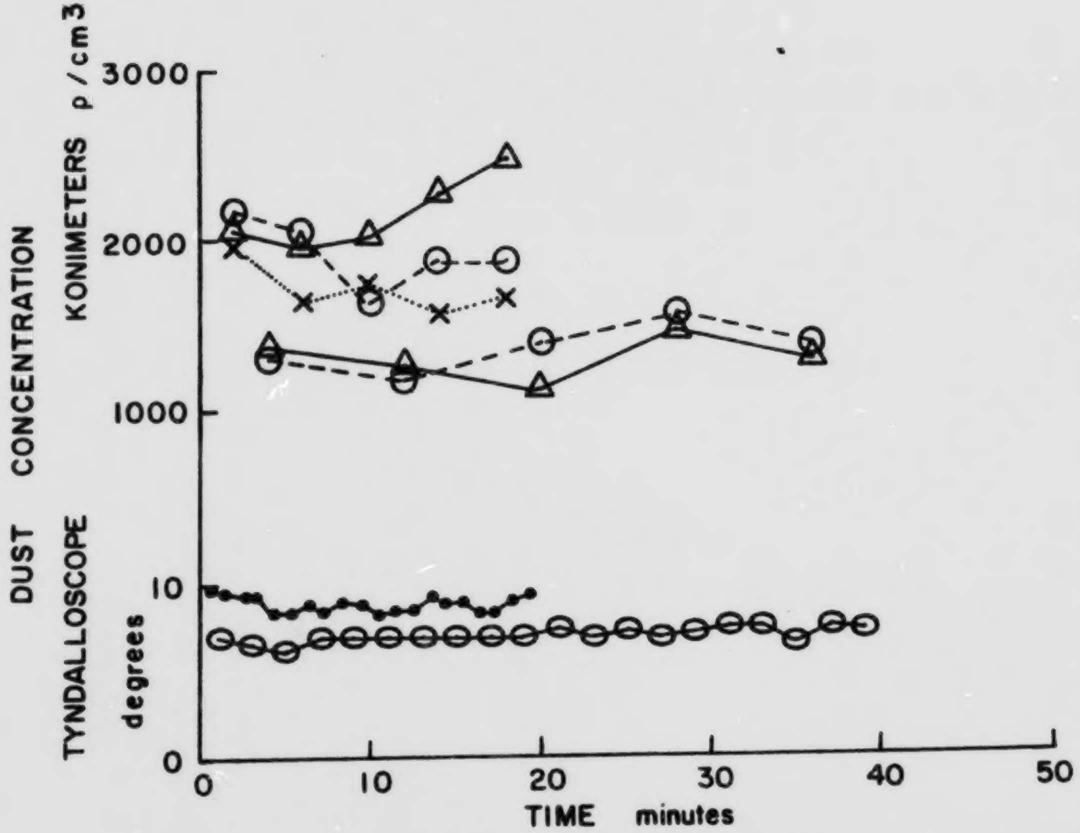
Material	Disperser	Size of Feed	Feed Rate cm^3/hour	Dust Concentration	
				p/cm^3	mg/m^3*
Coal	Air Ejector Pulveriser Jet Mill	Pulverised 45-100 mesh 45-100 mesh	20.5 20.5 2.88	310 880 400	2.5 4.0 1.6
Silica	Air Ejector Pulveriser Jet Mill	Pulverised 16-100 mesh 16-100 mesh	20.5 16.1 2.88	410 390 750	2.6 2.1 3.7
Pyrite	Air Ejector Pulveriser Jet Mill	Pulverised 100-200 mesh 100-200 mesh	20.5 20.5 0.56	250 440 430	2.7 2.9 3.0
Mica	Air Ejector Pulveriser Jet Mill	Pulverised 45-100 mesh 45-100 mesh	6.6 6.6 6.6	430 270 1050	4.0 2.5 7.4
Asbestos	Pulveriser Jet Mill	As Recd As Recd	6.6 2.88	370^F 950^F	2.7 1.7
Glass Fibre	Pulveriser Jet Mill	Chopped 1/8 x 1/8 Chopped 1/8 x 1/8	16.1 16.1	140^F 270^F	1.2 1.0

* respirable (Hexhlett)
F = fibres per cm^3



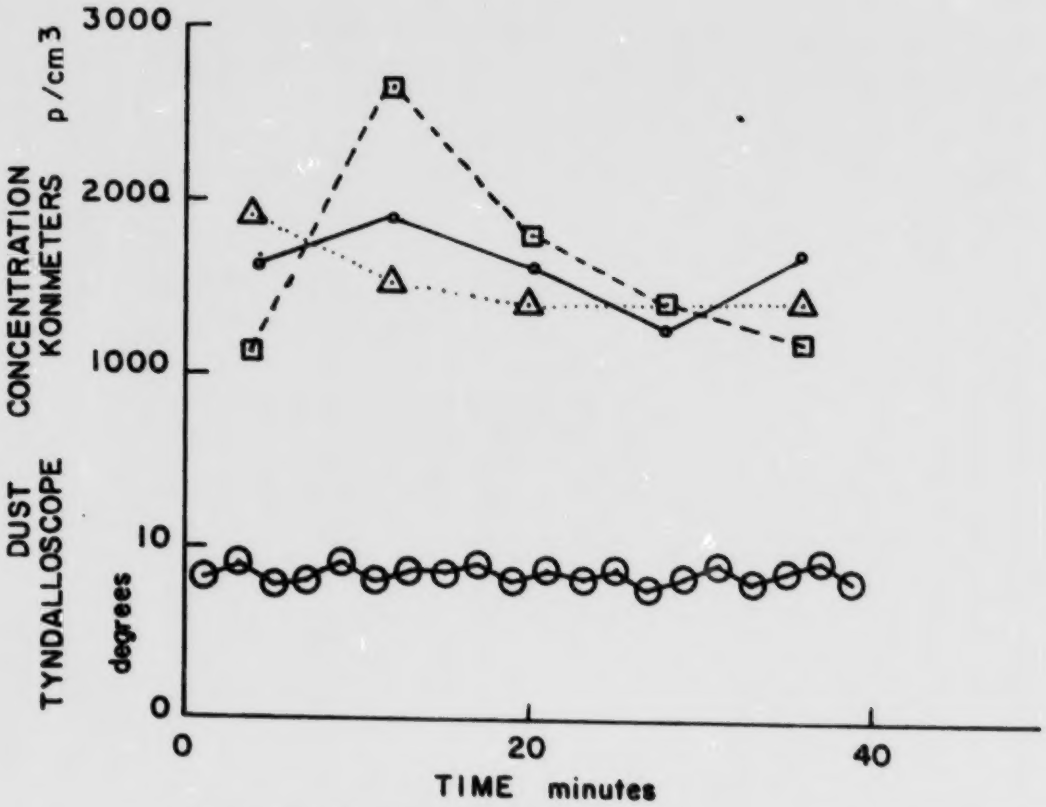
VARIATION IN DUST CONCENTRATION WITH TIME: 45 to 100-MESH COAL FED BY SMRE AND DISPERSED BY JET MILL

FIGURE A-4



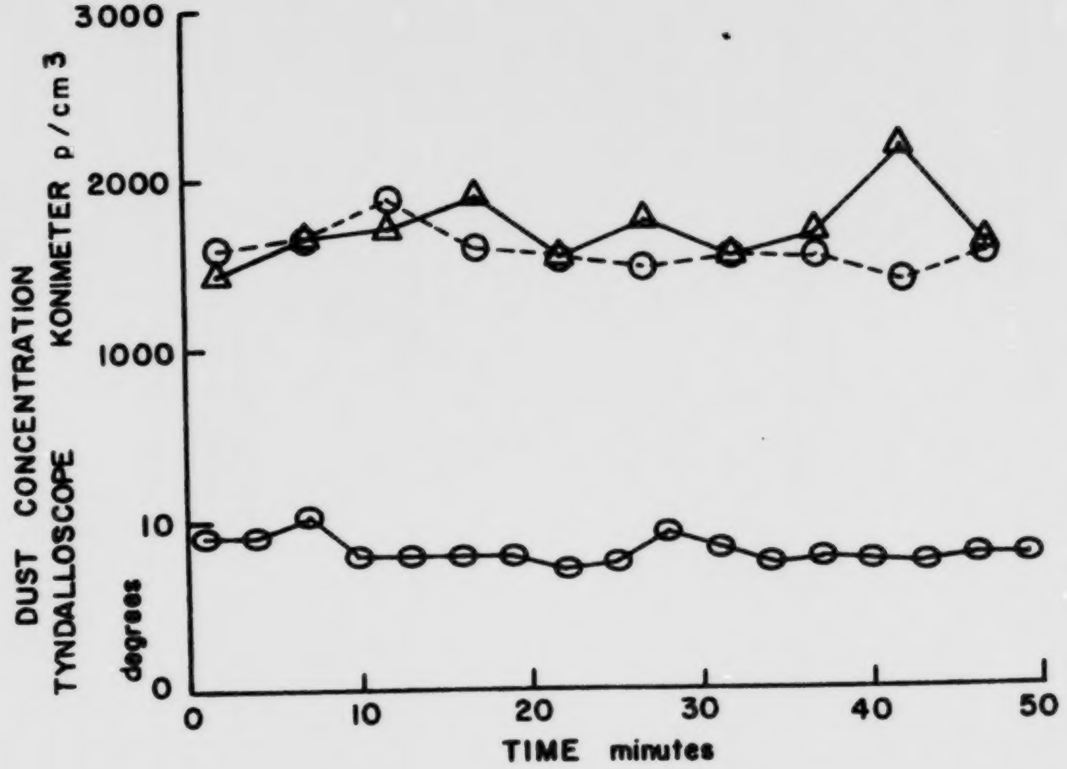
VARIATION IN DUST CONCENTRATION WITH TIME: PULVERIZED SILICA FED BY SMRE AND DISPERSED BY AIR EJECTOR

FIGURE A-5



VARIATION IN DUST CONCENTRATION WITH TIME: ASBESTOS FED BY SMRE AND DISPERSED BY JET MILL

FIGURE A-6



VARIATIONS IN DUST CONCENTRATION WITH TIME: PULVERIZED SILICA FED BY U.S.B.M. AND DISPERSED BY AIR EJECTOR

FIGURE A-7

great as the apparent variations with time; therefore, they are not necessarily as large as shown.

The difference in mean concentration for pairs of runs with the same dust at the same feed rate are shown in Table A4. The dust concentrations given were determined by the Hexhlett on a mass basis. It can be seen that the mean difference in dust concentration between two similar runs is about 10%. The results with other measures of dust concentrations, i. e., other instruments, are similar.

PARTICLE SHAPE

Some observations were made of the shape of dust particles after collection on thermal precipitator slides using a high-resolution binocular microscope. It was found that nearly all coal, silica, pyrite, and mica particles had length: breadth ratios less than 2. The thickness of small particles are difficult to determine on a microscope; however, it appeared that the breadth: thickness ratio of most of the coal, silica, and pyrite particles was less than 2 and that of most of the mica particles were greater than 4.

The asbestos and glass fibre dusts were a mixture of fibres and fine particles. The length:breadth ratio of the fibres was usually much greater than 3, the limit chosen to distinguish fibrous from non-fibrous particles. The asbestos particles frequently consisted of more than one fibre and many of the fine particles appear to be attached to the fibres.

The proportion of fibrous particles, expressed as

$$\frac{\text{Fibrous particles}}{\text{Fibres + non-fibrous particles} < 0.5\ \mu\text{m}} \quad \text{and}$$

present (5) in the four types of dust clouds produced from fibrous materials were: 0.77 in jet-milled asbestos,
0.20 in pulverized asbestos,
0.40 in jet-milled glass fibre,
0.46 in pulverized glass fibre.

TABLE A-4

Variation in Mean Dust Concentration Between Runs
(Hexhlett)

Material	Disperser	Concentration mg/m^3 Run 1	Concentration mg/m^3 Run 2	Ratio Large/Small
Coal	Jet Mill	1.64	1.38	1.19
	Air Ejector	2.28	2.48	1.09
Silica	Jet Mill	3.005	2.85	1.08
	Pulveriser	2.13	2.00	1.06
	Air Ejector	2.47	2.83	1.15
Pyrite	Jet Mill	3.72	3.78	1.02
	Pulveriser	2.83	2.91	1.03
	Air Ejector	7.43	6.31	1.18
Mica	Jet Mill	6.9	7.375	1.07
	Pulveriser	2.46	2.26	1.09
	Air Ejector	12.275	11.24	1.09
			Mean Value	1.10

SIZE DISTRIBUTION BY VOLUME: COULTER COUNTER

The results obtained for the dust clouds produced by the four dispersion methods from coal, silica, pyrite, and mica are shown in Figures A8 to A11. It can be seen that the departure from straight lines is small except near the toe, where large errors, due to the small number of large particles counted, can occur. The lines for the dust clouds from the four materials are nearly parallel except for the dust clouds produced by the jet mill from coal and silica, Figures A8 and A9. This may indicate that the breakage produced by the jet mill and the pulverizer is different for these two materials. Because the states of dispersion of these four materials, after they were prepared for the Coulter, were nearly complete and because only a few aggregates were seen on the thermal precipitator slides, the results are likely to be representative of the airborne dust clouds.

For the fibrous dust clouds, the results obtained by the Coulter Counter, Figures A12 and A13, are unreliable. There are two reasons for this: one, the state of dispersion; and two, the electrical response to a fibre passing through the orifice. Microscope examination shows that asbestos fibres are aggregated on the thermal precipitator slides and different results are obtained on the Coulter by different dispersion treatments; thus, the state of dispersion in the airborne dust cloud and in the Coulter electrolyte are most unlikely to coincide; this will render interpretation difficult. There is a large difference in the size distribution obtained using the two orifices for each of the fibrous dust clouds, whereas the difference between the orifices was within the accuracy of calibration for the other dust clouds. This difference leads to the suspicion that the Coulter Counter does not size long, thin particles or that there is an abnormal coincidence correction to be applied. Many of the fibres are longer than the zone of electrical sensing and must pass through the orifice aligned to the flow, thus only a portion of the fibre is in the sensing zone at any one time.

The values of the median size by volume and by number as well as the standard deviation for a log normal size distribution are given in Table A5.

SIZE DISTRIBUTION: OPTICAL MICROSCOPE

The size distribution of non-fibrous particles obtained by microscope examination of thermal precipitator slides were analyzed on a computer by a regression analysis technique (Appendix B). The results obtained are given in Table A6 for each of the twenty dust clouds. Further

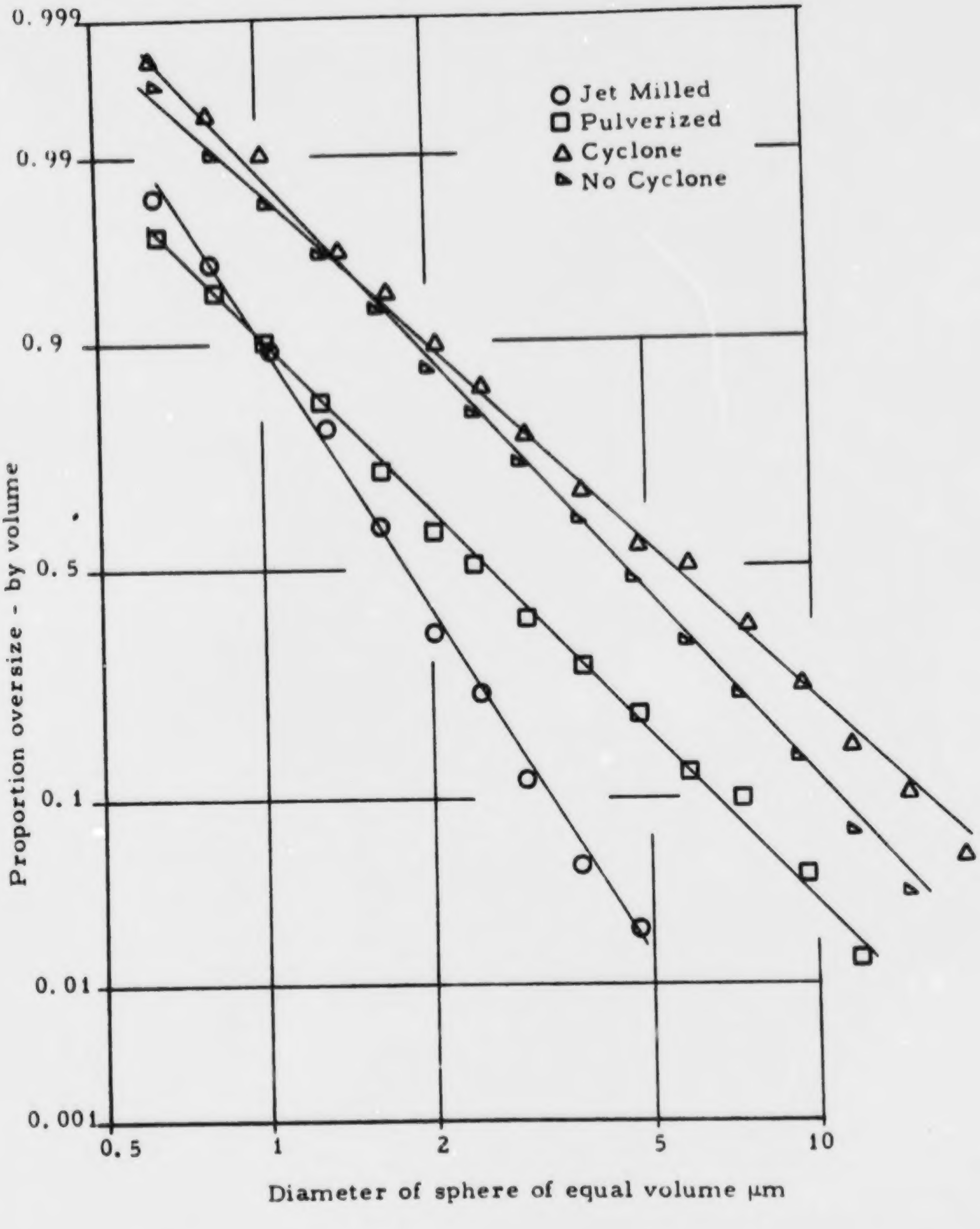


FIGURE A-8: Size distribution of the coal dust clouds - Coulter Counter.

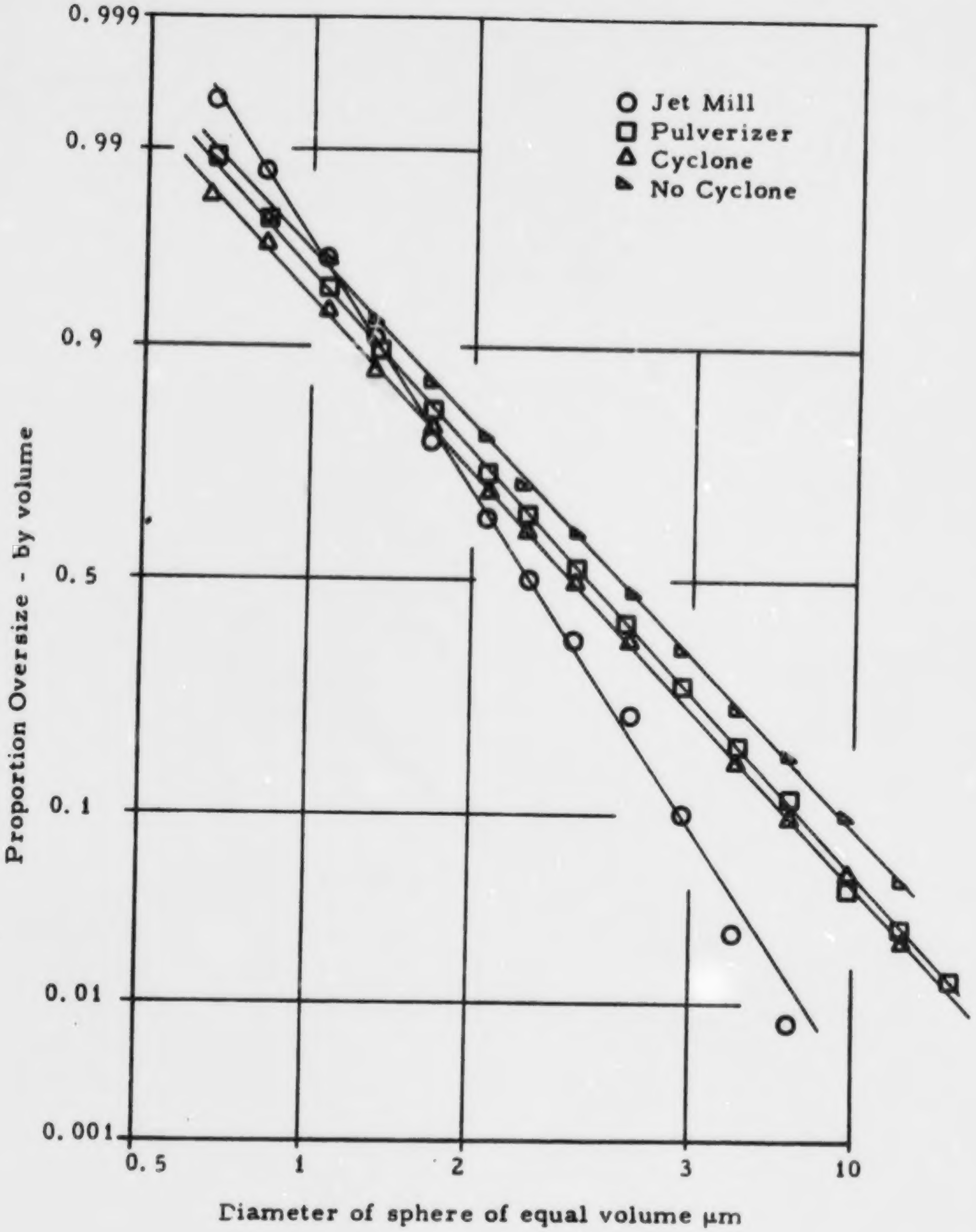


FIGURE A-9 Size distributions of the silica dust clouds - Coulter Counter.

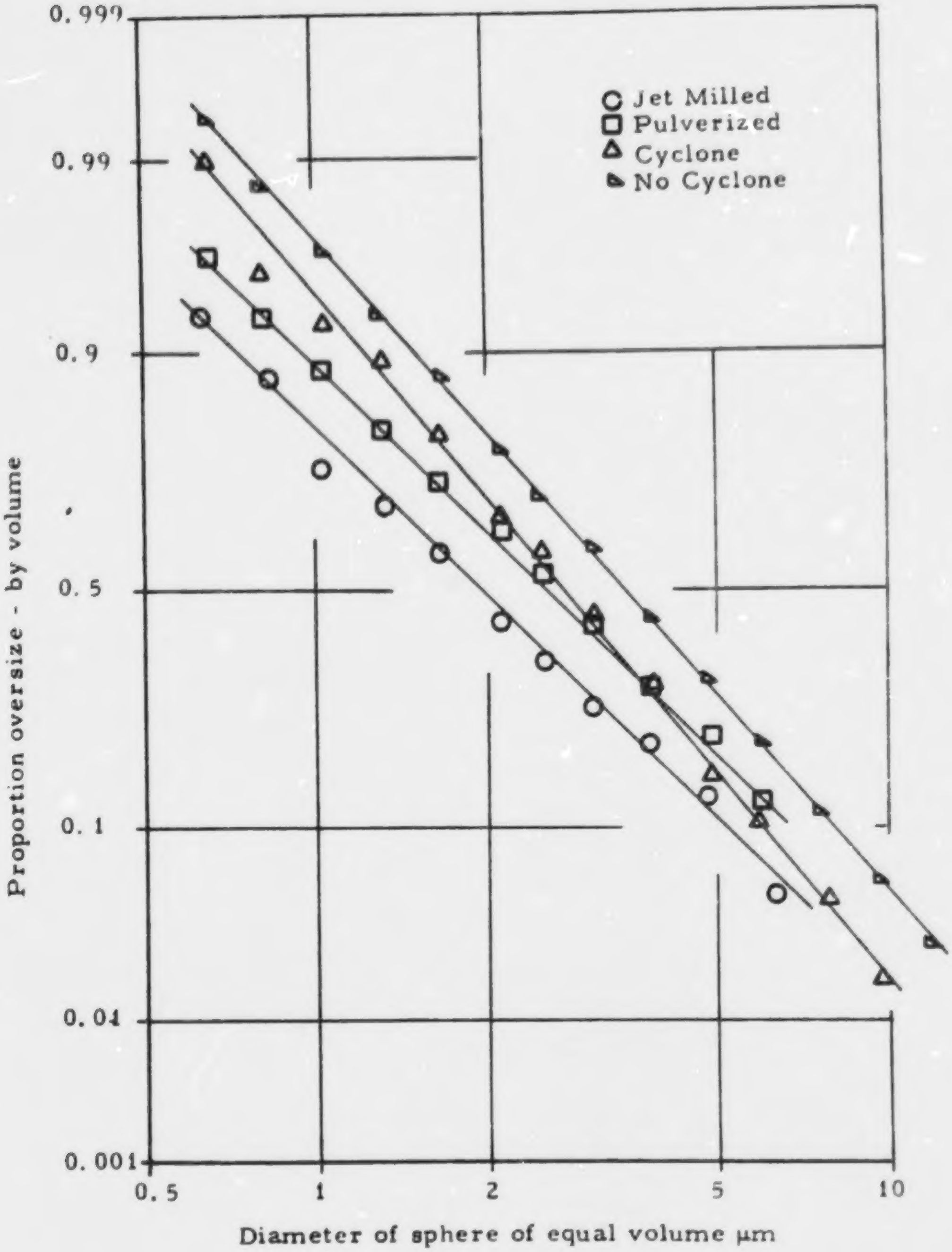


FIGURE A-10: Size distributions of pyrite dust clouds - Coulter Counter.

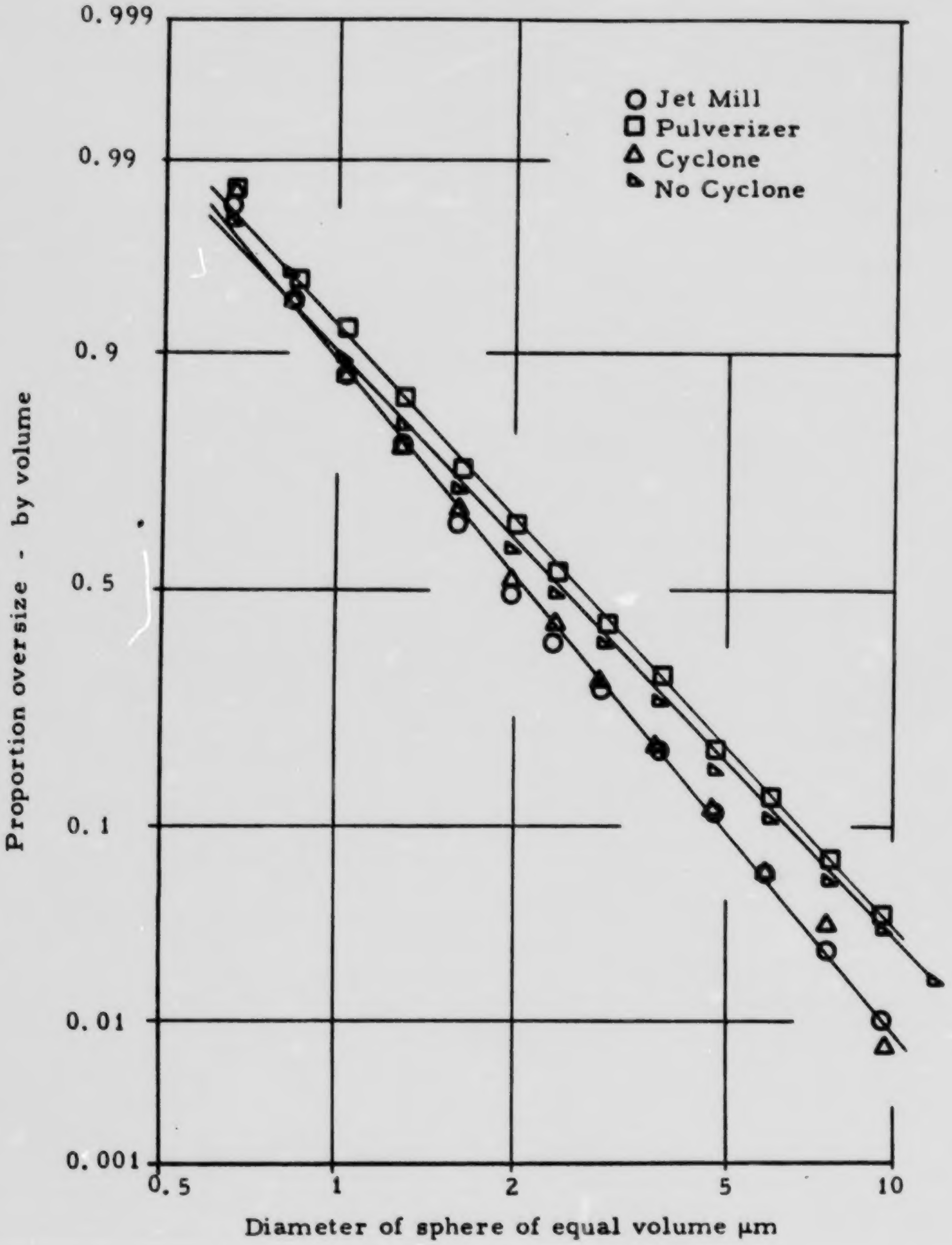


FIGURE A-11: Size distributions of the mica dust clouds - Coulter Counter.

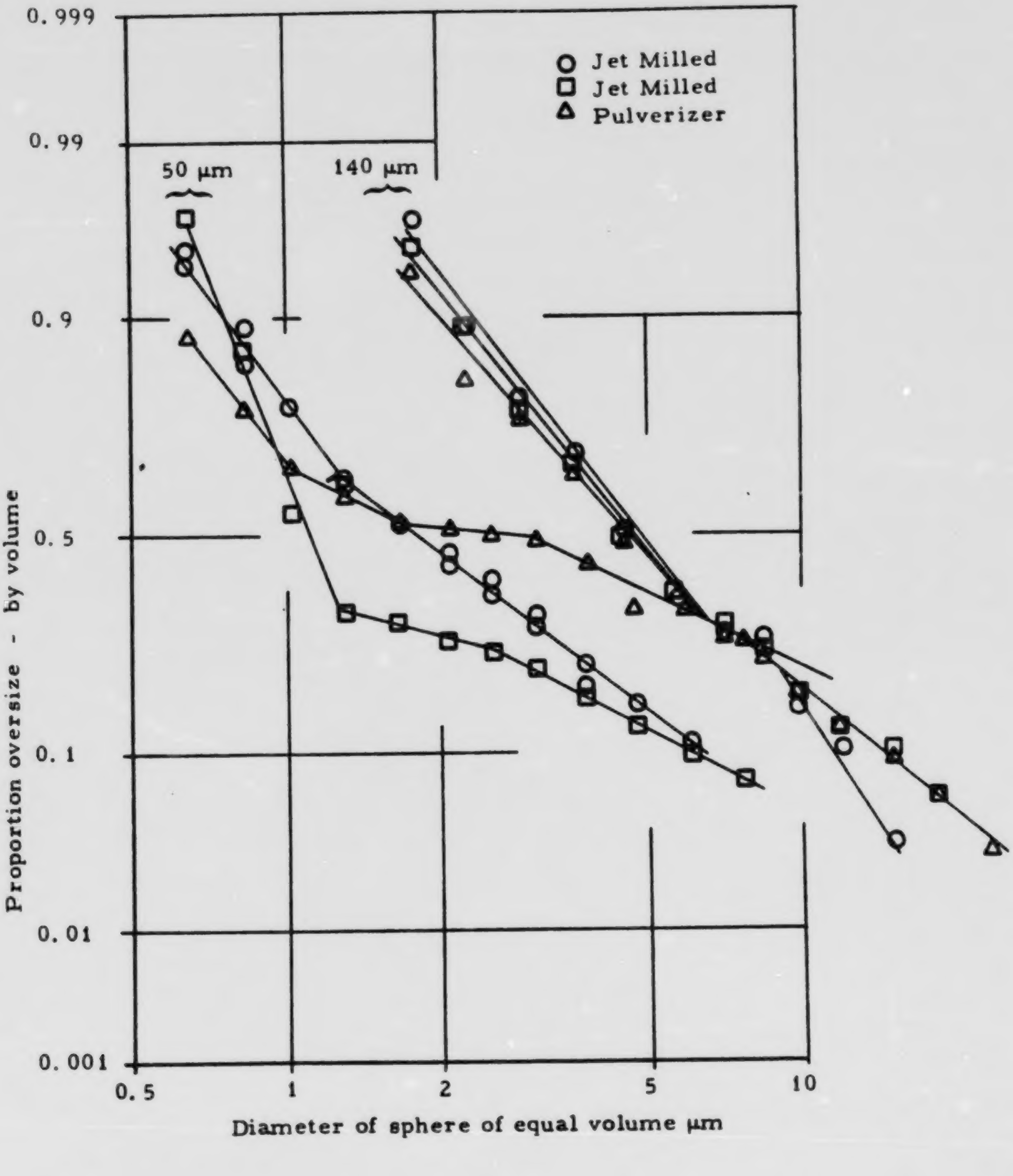


FIGURE A-12: Size distributions of asbestos dust clouds - Coulter Counter - 140- and 50-µm-diameter orifices.

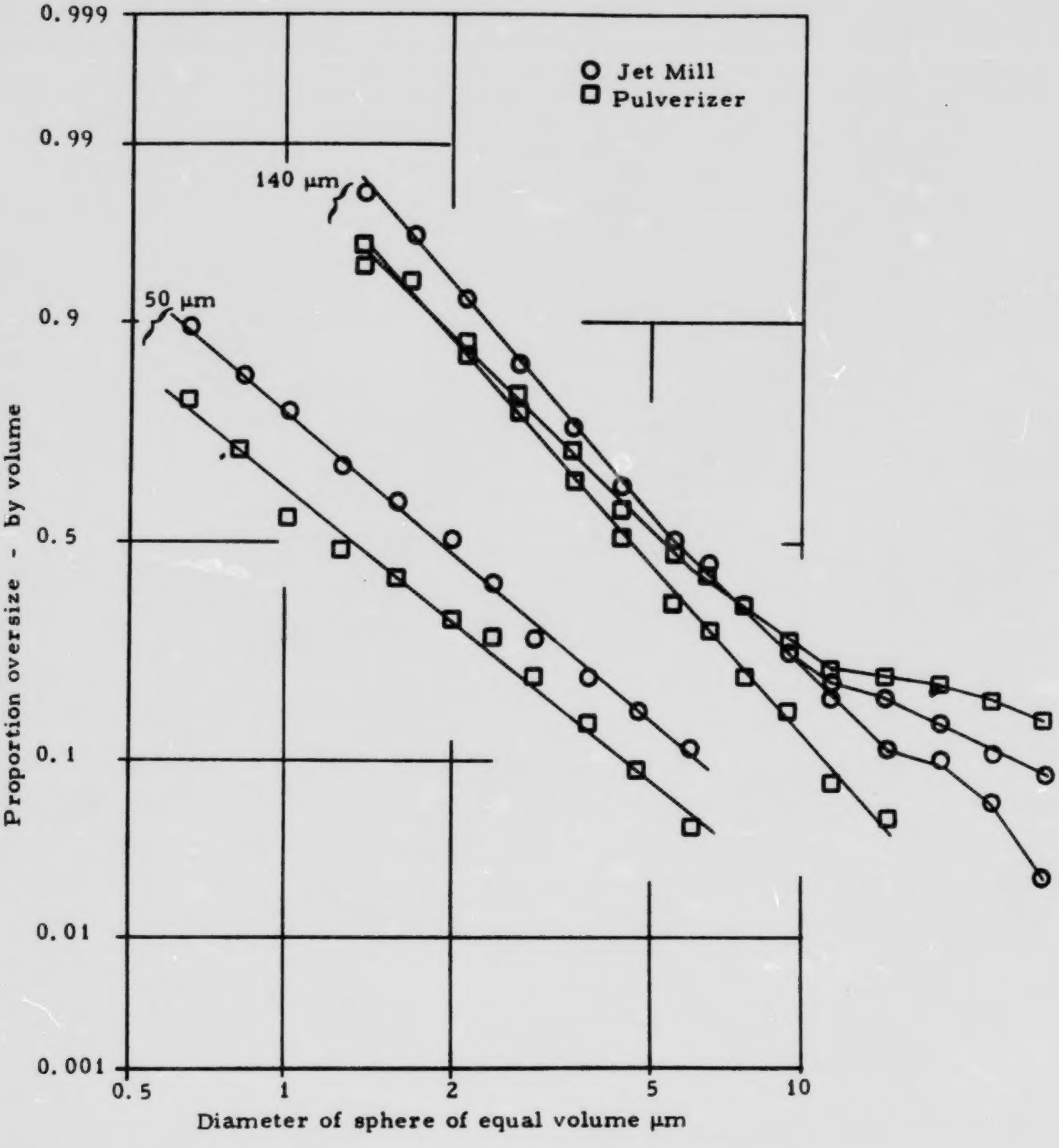


FIGURE A-13: Size distributions of the glass fibre dust clouds - Coulter Counter - 140- and 50-µm-diameter orifices.

TABLE A-5

Coulter Counter Log Normal Size Distributions Constants for Best Fit

Dust Cloud	Geometric Mean Size µm By Volume	By Number	Standard Deviation
Coal			
Jet Milled	1.8	1.0	1.6
Pulverised	2.6	0.52	2.1
Cyclone	4.5	1.15	1.95
No Cyclone	5.7	1.1	2.1
Silica			
Jet Milled	2.45	1.35	1.6
Pulverised	3.2	0.77	2.0
Cyclone	3.1	0.75	2.0
No Cyclone	3.7	0.89	2.0
Pyrite			
Jet Milled	1.9	0.29	2.2
Pulverised	2.6	0.48	2.15
Cyclone	2.8	0.82	1.9
No Cyclone	3.5	1.02	1.9
Mica			
Jet Milled	2.2	0.65	1.9
Pulverised	2.8	0.73	1.95
Cyclone	2.2	0.65	1.9
No Cyclone	2.6	0.55	2.05
Asbestos			
Jet Milled	1.1 - 4.6	—	2 - 5
Pulverised	2.7 - 4.8	—	2 - 5
Glass Fibre			
Jet Milled	2.1 - 5.9	—	2.6
Pulverised	1.3 - 5.5	—	1.9 - 2.65

TABLE A-6

Size Classification by Optical Microscope

Dust Cloud	Proportion of Dust in Size Range by Projected Area		
	<0.5 µm* / 0.5 µm	> 1/> 0.5 µm	> 5/> 0.5 µm
Coal			
Jet Milled	1.3	0.32	0.00014
Pulverized	1.1	0.38	0.008
Cyclone	0.25	0.607	0.0274
No Cyclone	-	0.609	0.0288
Silica			
Jet Milled	2.6	0.334	0.0041
Pulverized	0.35	0.351	0.0144
Cyclone	0.5	0.501	0.0113
No Cyclone	0.24	0.482	0.012
Pyrite			
Jet Milled	1.6	0.13	0.000066
Pulverized	5.1	0.1575	0.00249
Cyclone	0.35	0.509	0.00915
No Cyclone	-	0.526	0.0265
Mica			
Jet Milled	4.2	0.387	0.00735
Pulverized	2.1	0.262	0.00252
Cyclone	0.65	0.354	0.0138
No Cyclone	-	0.357	0.0166
Asbestos	Non Fibrous Particles Only		
Jet Milled		0.0445	0.018
Pulverized		0.167	0.0041
Glass Fibre			
Jet Milled		0.268	0.018
Pulverized		0.213	0.0103

* < 0.5 µm includes all particles less than 0.5 µm that are visible on a high-resolution microscope (light field, oil immersion objective, approx. N.A. of 1.0).

results for 'all visible' particles are also shown.

The diameters and lengths of fibrous particles have been determined on one thermal precipitator slide taken in each of the four types of fibre dust clouds. The ratios in the various size ranges obtained are shown in Table A 7.

TABLE A- 7
The Size Distribution in the Fibrous Dust Clouds

Dust Cloud	Fibres in Each Size Range Expressed as a Fraction						
	Diameter			Length			
	< 0.25 μm	0.25 - 0.5 μm	> 0.5 μm	< 5 μm	5 - 16 2/3	16 2/3 - 50	> 50 μm
Asbestos							
Jet Milled	0.68	0.17	0.15	0.72	0.26	0.02	0.0
Pulverized	0.70	0.16	0.14	0.52	0.38	0.07	0.03
Glass Fibre							
Jet Milled	0.16	0.65	0.19	0.60	0.30	0.04	0.06
Pulverized	0.20	0.60	0.20	0.39	0.52	0.09	0.0

It can be seen that the asbestos fibres are fine, many having diameters less than the resolving power of the microscope. The glass fibres are coarser than asbestos and the diameter of each fibre is greater than the resolving power of the microscope.

SIZE CLASSIFICATION BY FALLING SPEED

A dust sampler fitted with a horizontal elutriator size selector consists, in its usual form, of a number of rectangular channels through which air flows horizontally, followed by a filter paper to collect the 'respirable' dust. The airborne dust particles settle towards the floor of each channel. Some of these particles reach the floor and adhere.

The theory of horizontal elutriators was given by Walton(4) who showed that in the size range below a critical maximum size x_E (the design top-cut of the elutriator), the proportion of particles of a given size that settle out in a rectangular channel is proportional to their settling velocity.

The top size cut is that size corresponding to a particle that will just fall the height of the channel during the period of time required for the air to flow from one end of the channel to the other. Because the settling velocity is proportional to the square of the aerodynamic size, x, the proportion of size x, settling out is given by,

$$p = \frac{x^2}{x_E^2} \quad \text{for } 0 < x \leq x_E .$$

This theory was developed by Knight (5) to fit a mathematical size distribution to the measurements made by a number of dust samplers fitted with size selectors with different top cuts. The parameters of an assumed logarithmic normal size distribution obtained by this technique are shown for some of the dust clouds in Table A8, together with the classification of falling speed obtained for all the dust clouds in the main series of experiments is the ratio of respirable dust to total dust (open filter) by mass. The respirable dust being defined by the Hexhlett as fitted with a size selector.

SURFACE AREA

It is stated that silicosis is dependent on the surface area of the silica deposited in the lungs and thus efforts should be made to determine the surface area of the particles in the dust clouds and differentiate between the respirable fraction and the total dust.

The surface areas of the laboratory test dust clouds have been obtained by two methods:

1. from permeametric measurements;
2. calculated from the size distributions given by the Coulter.

The permeametric measurements were made on samples collected directly onto filter papers in the dust chamber. The techniques for calculation were made following three authors (6, 7, 8) and are given in Table A9. The surface areas obtained from the size distributions by volume are based on the two assumptions: 1, the size distribution is logarithmic normal; and 2, the particles are spheres.

TABLE A-8

Experimental Determinations of Particle Size Distributions Using Size Selective (Horizontal Elutriator) Samplers

Dust Cloud	Mass-Median Aerodynamic Size x_g μm	Geometric Standard Deviation σ_g	Respirable Dust Fraction by Mass
Coal			
Jet Milled	2.5	1.7	0.84
Pulverised	- -	- -	0.64
Cyclone	- -	- -	0.61
No Cyclone	4.9	2.0	0.39
Silica			
Jet Milled			0.74
Pulverised			0.47
Cyclone			0.57
No Cyclone			0.37
Pyrite			
Jet Milled	2.3	2.15	0.80
Pulverised	- -	- -	0.60
Cyclone	- -	- -	0.44
No Cyclone	7.4	2.0	0.24
Mica			
Jet Milled			0.89
Pulverised			0.83
Cyclone			0.71
No Cyclone			0.71
Asbestos			
Jet Milled	0.9	18.	0.64
Pulverised			0.74
Glass Fibre			
Jet Milled			0.28
Pulverised			0.60

TABLE A-9

The Specific Surfaces of Cloud Dusts

Dust Cloud	Size Collected	Porosity Fraction	Specific Surface Area m^2/cm^3			
			Kozeny Carmen	Arnell	Benarie	Calculated Coulter Counter
Coal						
Jet Milled	Total	0.66	43	53	13.3	30.1
	Respirable	0.76	67	82	12.1	
No Cyclone	Total	0.63	25	29	6.65	13.9
	Respirable	0.75	40	46	10.5	
Silica						
Jet Milled	Total	0.58	18.8	21	6.3	27.2
	Respirable	0.89	148	180	8.8	
No Cyclone	Total	0.57	21.5	25	8.1	21
	Respirable	0.81	76.5	93	11	
Pyrite						
Jet Milled	Total	0.79	84	150	13.8	43.5
	Respirable	0.84	141	193	16.6	
No Cyclone	Total	0.68	340	40	8.2	21.1
	Respirable	0.95	218	247	12.7	
Mica						
Jet Milled	Total	0.77	89	118	14.1	33
	Respirable	0.75	88	121	18.8	
No Cyclone	Total	0.72	61.6	77	13.4	30.3
	Respirable	0.80	93.5	118	13.5	
Asbestos						
Jet Milled	Total	0.86	100	118	12.3	54.2
	Respirable	0.87	120	143	12.1	

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APPENDIX B
STATISTICAL ANALYSIS TECHNIQUE

INTRODUCTION

About 7000 measurements of dust concentration were made during the first part of the program on Comparison of Dust Sampling Instruments in non-aggregated dust clouds, and this chapter discusses the statistical technique and the computer program used in analyzing the results.

The statistical analysis and computer program are based on the REGRE Program given in the IBM scientific subroutines. This is a multiple linear regression analysis program derived from a treatment by B. Ostle (2).

EXPERIMENTAL DESIGN FOR THE COMPARISON OF INSTRUMENTS

The experiments were laid out as a factorial design as described in most statistical textbooks. The factors were:

1. material - six levels - coal, silica, pyrite, mica, asbestos, and glass fibre;
2. dust dispersion method - four levels - jet mill, pulverizer, air ejector with, and without cyclone;
3. dust feed rate - three levels - feed rates were chosen for each dust cloud such that the levels differed by factors of 2 to 2 1/2 and that the lowest concentration was near the approved limit for silicosis.

A dust cloud prepared from one material by one method of dispersion is termed a "type of dust cloud", regardless of the feed rate or the concentration.

Because two of the methods of dispersion were not suitable for two of the types of dust, a total of twenty types of dust cloud were examined each at three concentrations in duplicate, thus producing a total of 120 dust clouds in which 7000 measurements of concentration were made by seventeen dust sampling instruments. Some instruments gave more than one estimate of the dust concentration, and they are arranged in groups measuring the same property of the dust.

METHOD OF STATISTICAL ANALYSIS

The problem in the statistical analysis is solving the expected relationship:

$$y = A \cdot F(x) \qquad \text{(Eq. B-1)}$$

where y and x are the dust concentrations estimated by the two instruments being compared. A is a constant; its value being dependent on the type of dust cloud in which the measurement is taken and F(x) is a function of x.

The expectation is that the relationship between y and x will be linear in most cases; however, the analysis should be able to detect non-linearity.

$$\text{Linear case} \quad F(x) = x \qquad \text{(Eq. B-2)}$$
$$\text{Non-linear} \quad F(x) = x^S \qquad \text{(Eq. B-3)}$$

An example of the dependence on the dust concentration of the differences between the dust concentrations estimated by three similar instruments is shown in Figure B-1, together with the effect of a logarithmic transformation on these errors. It can be seen that the errors in the linear form are greater at high concentrations than at low and that the logarithmic transformation smooths them out. This suggests that an equation of the form:

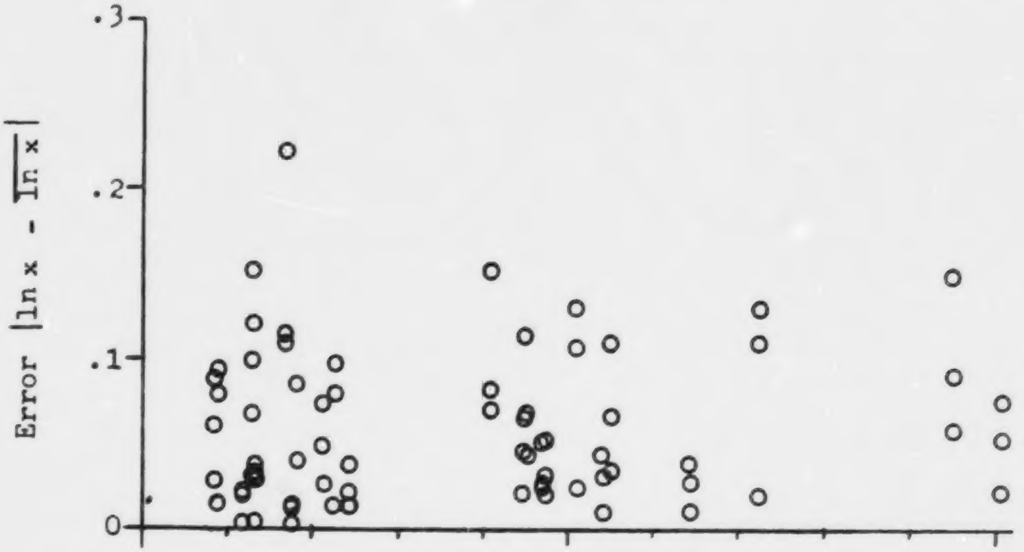
$$\ln y = B + C \ln x \qquad \text{(Eq. B-4)}$$

where ln y and ln x are considered as the variables while B and C are constants that might be applicable. The constant B in the above equation includes the differences between the various dust clouds and, if C is significantly different in value from 1, it would indicate non-linearity. Factor levels such as type of dust cloud, which have no numerical significance, can be handled by using dummy variables (3) which take the value 1 if the measurement is taken at the appropriate factor level and 0 if it is not.

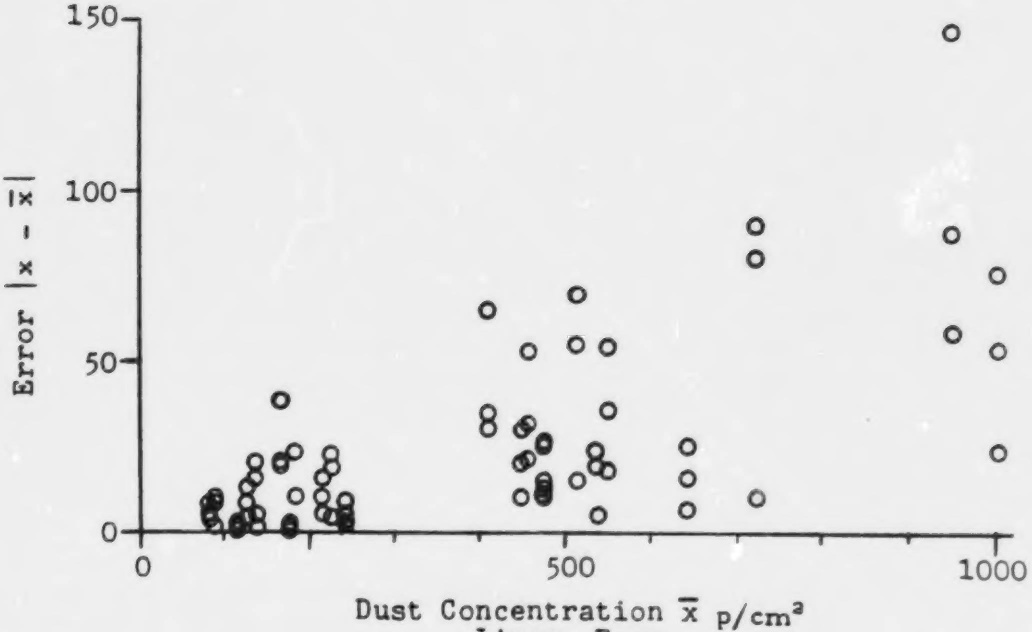
By using dummy variables, the constant B which depends on the type of dust cloud or characteristics of the individual sampling instrument in each group can be expanded to give an equation suitable for statistical and computer analysis:

$$\ln y = b_0 + b_1 \ln x + b_2 X_2 + \ldots + b_m X_m + b_{m+1} X_{m+1} + \ldots b_n X_n \qquad \text{(Eq. B-5)}$$

where, y = the concentration measured by the test instrument,
x = that measured by the reference instrument,
$\ln y$ = the dependent variable,
$\ln x$ = the independent variable,
b_0 = the intercept on the logarithmic plot,



Logarithmic Transformation



Linear Form

FIGURE B.1 THE DEPENDENCE OF THE ERRORS ON THE DUST CONCENTRATION (THE ERROR GIVEN IS THE DIFFERENCE BETWEEN EACH IMPINGER AND THE MEAN OF THREE IN EACH DUST CLOUD).

b_1 to b_n = the regression coefficients,
X_2 to X_n = the dummy independent variables.

This regression equation can be divided into various sections as shown below:

1. the dependent variable ln y;
2. the intercept b_0;
3. the feed rate or reference instrument part b_1 ln x;
4. the part containing the dummy variables pertaining to the different dust clouds - $b_2 X_2$ to $b_m X_m$;
5. the part containing the dummy variables pertaining to the different test instruments in each analysis: $b_{m+1} X_{m+1}$ to $b_n X_n$.

These will, where necessary, be amplified in the following sections.

The Reference Instrument

The original intention in this analysis was to use the feed rate as an independent variable. However, a feed rate is not reproducible or measurable with sufficient accuracy, and a given feed rate results in a very wide range of concentrations depending on material and method of dispersion. Therefore each concentration (y) measured by the instrument under "test" has been compared with that (x) measured by a "reference" instrument in the same dust cloud. The program has been set up so that the log (concentration) measured by one instrument or the mean of the log measured by a group of instruments, i. e., the three thermal precipitators, can be used as the reference.

Independent Variables for Types of Dust Cloud

It seemed simpler to carry out the analysis with twenty types of dust cloud, and then determine the effects due to material and method of dispersion, rather than have five variables for interaction. In the analysis, as the measurement must have been made in one and only one of the twenty types of dust cloud, we have:

$$X_1 + X_2 + \dots + X_{20} = 1 \qquad \text{(Eq. B-6)}$$

where X_1 to X_{20} = the dummy variables taking values of 0 or 1 and nineteen independent variables are sufficient to specify in which of the twenty types of dust cloud the measurement was taken. If a twentieth is used it produces an uncertainty and the equation for the regression coefficients would not be solvable. Inserting these nineteen dummy variables into the equation leads to the derivation of nineteen regression coefficients. The omission of the twentieth dummy variable is equivalent to setting the regression coefficient to 0. The regression coefficients (b) then measure the departure of the mean of each dust cloud from that of the dust cloud for which the dummy variable was omitted. The departure (B) of each dust cloud from the overall mean would be more valuable, and these can be derived as shown:

$$b_i = B_i - B_m \quad i = 1, \ldots, m-1 \qquad \text{(Eq. B -7)}$$

$$b_m = 0 \qquad \text{(Eq. B-8)}$$

Hence,

$$\sum_{i=1}^{m-1} b_i = \sum_{i=1}^{m-1} B_i - (m-1)\, B_m \qquad \text{(Eq. B-9)}$$

$$= -B_m - (m-1)\, B_m \quad \left(\text{since } \sum_{i=1}^{m} B_i = 0\right) \qquad \text{(Eq. B-10)}$$

$$= -\, m\, B_m \qquad \text{(Eq. B-11)}$$

therefore,

$$B_m = \frac{1}{m} \sum_{i=1}^{m-1} b_i \qquad \text{(Eq. B-12)}$$

therefore,

$$B_i = b_i - \frac{1}{m} \sum_{i=1}^{m-1} b_i . \qquad \text{(Eq. B-13)}$$

The meaning of these coefficients can be clarified by considering the ith dust cloud.

The ith dummy variable takes the value 1 and the rest of the dust cloud dummy variables are 0, thus,

$$\log y = B_0 + b_1 \log x + B_i \cdot 1.0 + B_j \cdot 0.0 \qquad \text{(Eq. B-14)}$$

where B_0 = the corrected intercept

or

$$y = x^{b_1} \cdot e^{B_0} \cdot e^{B_i} = x^{b_1} \cdot e^{B_0 + B_i} \qquad \text{(Eq. B-15)}$$

and exp (B_i) is the factor by which the overall mean is to be multiplied to obtain the mean for the ith dust cloud.

The mean effect for each material and each method of dispersion can be found similarly as shown below. Let t_1 be the mean value (over the four methods of dispersion) of the coefficients of departure obtained on the first material, and t_2 that on the second, etc. then

$$t_1 = \frac{1}{4} \sum_{i=1}^{4} B_i \,, \quad t_2 = \frac{1}{4} \sum_{i=5}^{8} B_i \,, \quad \text{etc.} \qquad \text{(Eq. B-16)}$$

The desired coefficient of departure T_i from the overall mean is obtained by setting

$$\sum_{i=1}^{6} T_i = 0 \qquad \text{(Eq. B-17)}$$

thus,

$$T_i = t_i - \frac{1}{6} \sum_{j=1}^{6} t_j \qquad \text{(Eq. B-18)}$$

similarly for the coefficients of departure for method of dispersion.

These results are printed by the program as:

1. the regression coefficients with an estimate of their standard errors;
2. a table of coefficients of departure from the overall mean for individual types of dust cloud, for mean material and for mean method of dispersion;
3. anti logarithms of the coefficients in 2 are also printed; these are multiplying factors for the ratios of dust concentrations.

Independent Variables for Instruments

Groups of instruments measuring dust concentration in the same way are analyzed together, i.e., one group is the staplex, electrostatic precipitator and the open filter all measuring the total mass of dust in the air, and the second group is the konimeters. Each instrument but one in a group is assigned a dummy independent variable, of value 1.0 or 0.0. The resultant regression coefficients indicate the mean differences between the corresponding instruments and the instrument for which the dummy variable was omitted. Each instrument provides a measurement of the dependent variable.

The Intercept

. The intercept b_0 represents the value of the dependent variable when all the other variables are 0;

that is, $\ln y = b_0 + b_1 \log x + b_2 X_2 + \ldots + b_N X_N$ (Eq. B-19)

where $\ln x = 0$ and X_2 to $X_N = 0$,

or, $\ln y = b_0$ when $x = 1$. (Eq. B-20)

Therefore, the ratio of dust concentrations from test and reference instruments is given by,

$$\ln y - \ln x = b_0 \quad \text{or} \quad \frac{y}{x} = e^{b_0} \qquad \text{(Eq. B-21)}$$

This ratio pertains to the mean value of the particular dust cloud and instrument for which the dummy variables were omitted, and the corrected value for the mean over all the dust clouds and instruments is,

$$\ln \left(\frac{y}{x}\right) = B_0 = b_0 + k_D + k_I \qquad \text{(Eq. B-22)}$$

where the k's are the correction factors determined earlier.

If the relationship between the dust concentrations from the test and reference instruments is not linear (i.e., b_1 is not equal to 1), the ratio of concentrations varies with the concentration and the intercept B_0 is the estimated value at $x = 1$. As the mean value of x can be much greater than 1, a better estimate of the mean ratio $\frac{y}{x}$ can be found from the mean values of ln y and ln x by:

$$\sum_{i=1}^{N} \ln \frac{y_i}{x_i} = \sum_{i=1}^{N} \ln y_i - \sum_{i=1}^{N} \ln x_i \qquad \text{(Eq. B-23)}$$

where N = the total number of observations, and
i = the serial number of the individual observations.

The intercept, the corrected intercept, the mean ratio (from the corrected intercept) and the mean ratio (from the logarithmic means) are given by the program.

THE COMPUTER PROGRAM

The computer program is based on the IBM scientific subroutines REGRE, CORRE, ORDER, MINV and MULTR which analyze data and perform a multiple linear regression with up to forty variables. These subroutines are run under a main program called "DUSTMLR" which prepares the data for each problem into a form suitable for the other subroutines.

The functions of the various sections of the computer program are outlined below:

DUSTMLR

1. reads dust concentrations in batches of 120, each batch consisting of one measurement on each dust cloud by one instrument;
2. prints all dust concentrations for verification;
3. reads parameter card for each problem; this defines test and reference instruments;
4. reads dust cloud selection card ;
5. selects each measurement made by the "test" instruments over the required range and the corresponding measurements by the "reference" instruments and takes their logarithms;
6. calculates the values of the dummy variables pertaining to each test instrument measurement ; and
7. writes the values of the variables on tape in a form suitable for CORRE.

REGRE

8. reads the dependent and independent variable selection card;
9. calls the remaining subroutines in order;
10. calculates the variance attributed to the feed rate (or reference instrument), the dustclouds and the test instruments;
11. calculates the tables of dust type, method of dispersion, and dust cloud coefficients and multiplying factors;
12. prints all answers; and
13. prints the residual from both multiple regression and simple linear analysis for each observation (test and concentrations); optional.

CORRE

14. calculates means, standard deviations, and sums of cross products of deviations from means and product moment correlation coefficients from the data placed on tape by DUSTMLR.

ORDER

15. selects the variables to be analyzed;
16. prepares a matrix of intercorrelations among independent variables;
17. prepares a vector of intercorrelations of independent variables with the dependent variable.

MINV

18. inverts the matrix of intercorrelations among the independent variables.

MULTR

19. calculates regression coefficients;
20. determines the intercept b_o;
21. computes the multiple correlation coefficient;
22. performs analysis of variance; and
23. certain other statistics are computed - standard deviations of regression coefficients, computed t values of regression coefficients.

PRESENTATION OF DATA AND CONTROLS

The input data is punched on cards using four significant figures and an exponent (E 6.3 specification). This is arranged in sets of 120 measurements on twelve punch cards. Each set consists of one type of measurement made by one instrument for each dust cloud.

Three control cards are used:

1. DUSTMLR Control Card - specifies the test and reference instruments, the number of selections and the number of variables;
2. DUSTMLR Selection Card - controls the selection of measurements from specified types of dust clouds; and
3. REGRE Selection Card - is used to specify the dependent variable and the set of independent variables for the regression analysis.

PRESENTATION OF RESULTS

The program repeats the parameter cards and prints the problem number for each problem.

The printout for each problem consists of the following

1. mean of each variable;
2. standard deviation of each variable;
3. correlation coefficient between each independent variable and the dependent variable;
4. regression coefficients;
5. standard errors of the regression coefficients;
6. computed t values;
7. intercept;
8. multiple correlation coefficient;
9. standard error of estimate - this term arises in the original IBM program, but the calculation would seem to produce the similar "standard error of observation";
10. corrected intercept;
11. mean ratio of test to reference dust concentrations and its geometric error;
12. an analysis of variance;
13. table of coefficients of departure from the overall mean for each dust type, size (method of dispersion) and dust cloud;
14. table of multiplying factors as for 13;
15. table of each observation of dependent variable and its residual after multiple and single regression analysis - optional.

The results of a typical analysis are given below.

Printout from Computer Program showing a Comparison between Thermal Precipitators and Impingers

```
PROBLEM NUMBER TP13MI         NUMBER OF OBSERVATIONS    335
DUSTCLOUDS       1 TO    20 NUMBER OF VARIABLES 23
VARIABLE        MEAN       STANDARD       CORRELATION
  NO.                      DEVIATION         X VS Y          REGRESSION
                                                             COEFFICIENT
  2  REF I   5.84464       1.08347        -0.17616          -0.13403
  3  ↑        .04776        .21358          .20422            .11448 ⎫
  4  |        .05075        .21981        -0.07481          -0.53698 ⎬ Coal
  5  |        .04776        .21358        -0.08679          -0.56661 ⎭
  6  |        .05373        .22582          .08354          -0.13914 ⎫
  7  |        .05373        .22582        -0.00553          -0.37051 ⎬ Silica
  8  T        .05373        .22582        -0.17237          -0.72992 ⎪
  9  Y        .05373        .22582        -0.14795          -0.67540 ⎭
 10  P        .05373        .22582          .08192          -0.05108 ⎫
 11  E        .05373        .22582          .26510            .23209 ⎬ Pyrite
 12  S        .05373        .22582          .04058          -0.34079 ⎪
 13           .04776        .21358          .01146          -0.38501 ⎭
 14  O        .05373        .22582        -0.06829          -0.39163 ⎫
 15  F        .05373        .22582          .08826          -0.11293 ⎬ Mica
 16           .05373        .22582        -0.17556          -0.60413 ⎪
 17  D        .05373        .22582        -0.15512          -0.57205 ⎭
 18  U        .04478        .20712          .09237          -0.16579 ⎫ Asbestos
 19  S        .05373        .22582        -0.09227          -0.51454 ⎭
 20  T        .04776        .21358        -0.15066          -1.03985 ⎫ Glass
 21. ↓        .01493        .12144          .15983            .06427 ⎭
 22  INSTR    .33731        .47350          .03404            .12195 LPDS head
 23  VARIA    .32239        .46809          .11164            .20030 LPDS
DEPENDENT
  1          1.07833        .52391
                                        STD. ERROR         COMPUTED
                          Variable N    OF REG.COEF.        T VALUE
                               2           .03040          -4.40902
                               3           .14438            .79294
REF I = Reference Instrument   4           .14200          -3.78156
                               5           .14429          -3.92693
INSTR                          6           .14084          -0.98790
VARIA = Instrument Variables   7           .14011          -2.64445
                               8           .14021          -5.20576
                               9           .14023          -4.81645
                              10           .14478          -0.35278
                              11           .14024           1.65496
                              12           .14006          -2.43321
                              13           .14418          -2.67026
                              14           .14417          -2.71651
                              15           .14131          -0.79916
                              16           .14552          -4.15152
                              17           .14474          -3.95216
                              18           .14684          -1.12902
                              19           .14114          -3.64555
                              20           .15884          -6.54654
                              21           .21891            .29357
                              22           .05573           2.18800
                              23           .05648           3.54643
```

Printout .... (2)

```
TOTAL OF DUST TYPE SIZE REGRESSION COEFFICIENTS      -6.78552

INTERCEPT                        2.10577

MULTIPLE CORRELATION              .63322

STD. ERROR OF ESTIMATE            .41955


CORRECTED INTERCEPT              1.87660

MEAN RATIO OF CONCENTRATIONS FROM TEST

                  AND REFERENCE INSTRUMENTS BASED ON

   CORRECTED INTERCEPT           6.5312469
   LOGARITHMIC MEANS              .0065118
GEOMETRIC ERROR OF RATIO         1.52127



                       ANALYSIS OF VARIANCE FOR THE REGRESSION

    SOURCE OF VARIATION         DEGREES     SUM OF    MEAN       F VALUE
                               OF FREEDOM  SQUARES   SQUARES
ATTRIBUTABLE TO FEED RATE            1      2.84500   2.84500  16.16303
ATTRIBUTABLE TO INSTRUMENTS          2      2.26044   1.13022   6.42101
ATTRIBUTABLE TO DUST CLOUD TYPES    19     31.65495   1.66605   9.46519
ATTRIBUTABLE TO REGRESSION          22     36.76038   1.67093   9.49289
DEVIATION FROM REGRESSION          312     54.91784    .17602
      TOTAL                        334     91.67823
```

Printout ....(3)

```
                         TABLE OF DUST TYPE-SIZE FACTORS

                    DUST SIZE MEAN        COAL          SILICA

       DUST TYPE MEAN,                  .1044512      -0.1270166
       JET MILLED          .0767104     .3392759       .2001398
DUST   PULVERISED          .2767659     .4537593      -0.0312376
       CYCLONE           -0.1783308   -0.1977010      -0.3906432
SIZE   NO CYCLONE        -0.1751456   -0.2273317      -0.3361276

         PYRITE            MICA         ASBESTOS      GLASS FIBRE

       DU T M  .2155295  -0.0684585     .0115602      -0.1360658
       JET     .2882000  -0.0523509     .1734872      -0.7005753
       PULV    .5713673   .2262463    -0.1752679       .4035424
       CYC   -0.0015158  -0.2648536        0               0
       NO CY -0.0457358  -0.2327778        0               0

 MULTIPLYING FACTORS FOR DEPARTURE OF CONCENTRATIONS
                                             FROM OVERALL MEAN
                                        COAL          SILICA
       DUST TYPE MEAN,                1.1101012       .8807191
       JET MILLED        1.0797294    1.4039307      1.2215735
DUST   PULVERISED        1.3188576    1.5742191       .9692453
       CYCLONE            .8366656     .8206152       .6766216
SIZE   NO CYCLONE         .8393348     .7966565       .7145320
         PYRITE            MICA        ASBESTOS      GLASS FIBRE
       DU T M 1.2405186    .9338322   1.0116273       .8727852
       JET    1.3360240    .9489958   1.1894454       .4962997
       PULV   1.7706865   1.2540098    .8392321      1.4971188
       CYC     .9984853    .7673183       0              0
       NO CYC  .9552943    .7923296       0              0

       DU T M = Dust Type Mean
```

DISCUSSION

The validity of the regression analysis depends on meeting the assumptions on which the method of least squares is based. The assumptions (4) are:

1. the average value of the errors is 0;
2. the errors have common variance;
3. the errors are independent;
4. the values of the independent variables are measured with negligible error.

For tests of significance the errors are also assumed to follow a normal distribution. Figure B-2 shows that the residuals in these analyses are close to a normal distribution.

Departure from the first assumption could be compared to a systematic difference between the counting of dust samples between laboratories, and thus, accepting the assumption is equivalent to saying that the results do not take into account the difference between this laboratory's estimate and a "true" mean of all laboratories.

The instruments are compared by subjecting them to the same dust cloud. They thus experience, except for variation from point to point in the chamber, the same fluctuations in concentration and the same mean concentration, which they are intended to measure. Provided that both samples are large enough, the deviation of each measurement from the "true" mean value should be approximately normally distributed at one concentration. These are the deviations that affect the analysis and the deviations of the "true" concentrations from the set or intended values are largely immaterial. Thus the errors are independent, and, after a logarithmic transformation as shown on Figure B-1, are of common variance.

The Errors of the Independent Variables

The independent variables can be divided into three groups:

1. the reference dust concentration;
2. the dummy variables pertaining to the materials;
3. the dummy variables pertaining to the instruments in the "test" group.

It is clear that the errors in the measurement of the reference dust concentration are of the same order as that of the test dust concentration

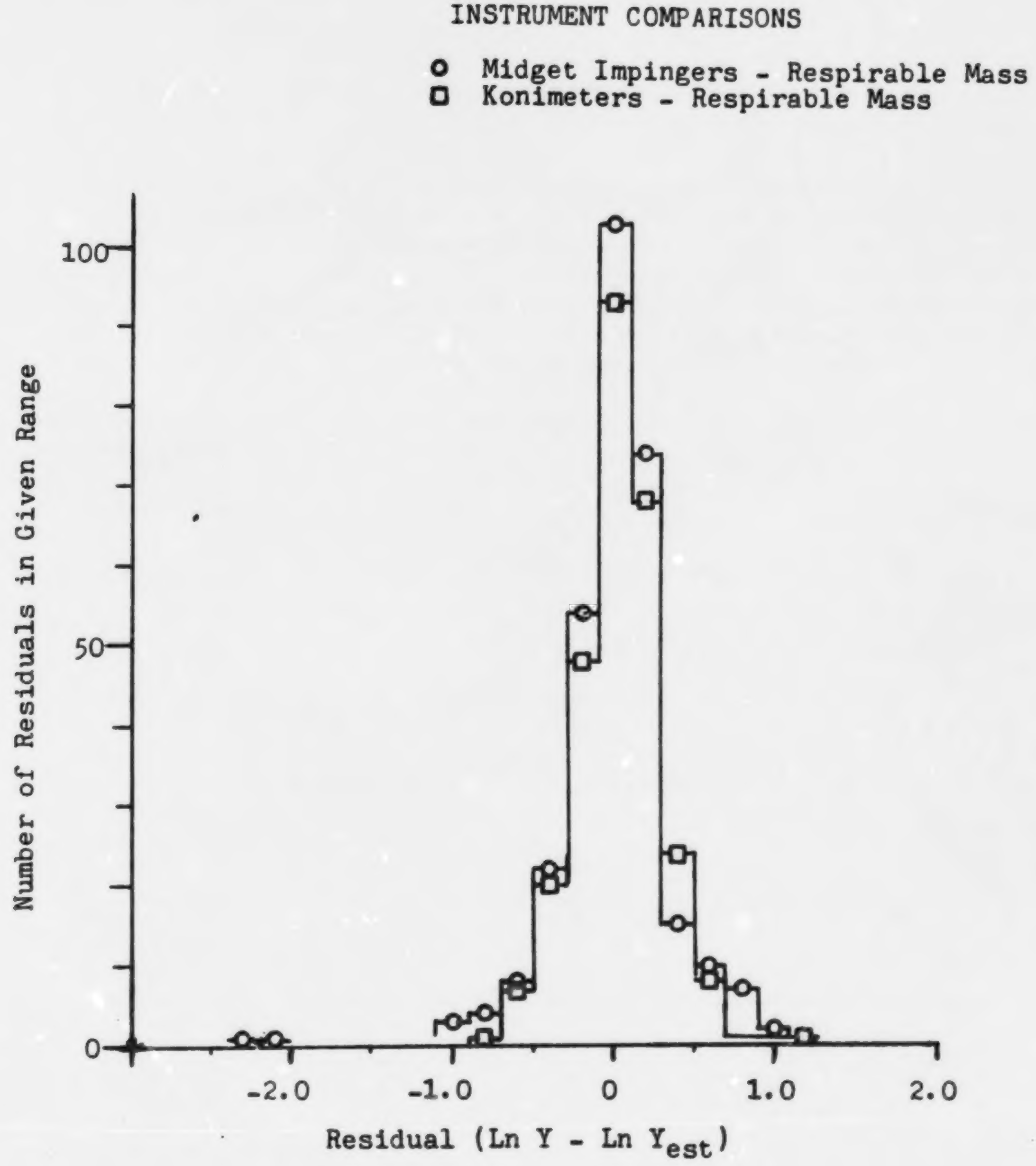


FIGURE B.2 DISTRIBUTION OF RESIDUALS FROM TWO REGRESSION ANALYSES (Eq. B.3).

(the dependent variable). However, the errors are small compared to the range of concentrations examined.

No errors are associated directly with the dummy variables. But variations in dust size distribution, etc. between the six dust clouds of one type could be considered equivalent to an error in the dummy variable; however, as these errors are small compared to the differences between types of dust cloud and to those of the dependent variable, they are assumed to have no effect on the analysis (5). There seem to be no errors associated with the dummy variables pertaining to the instruments.

The major departure from the assumption for the least squares method is the magnitude of the errors of the independent variable, reference dust concentration, in relation to that of the dependent variable. To examine this further, some comparisons have been examined in a number of ways.

Firstly, the values of the coefficients in the regression equation were determined.

$$\ln y = b_0 + b_1 \ln \bar{x} + b_2 X_2 + b_3 X_3 + \ldots b_n X_n \qquad \text{(Eq. B-24)}$$

Then those in the corresponding equation with the test and reference instrument transposed:

$$\ln x = b_0' + b_1' \ln \bar{y} + b_2' X_2 + \ldots + b_n' X_n \qquad \text{(Eq. B-25)}$$

This can be rearranged so that the terms match those in Equation B-24.

$$\ln y = -\frac{b_0'}{b_1'} + \frac{1}{b_1'} \ln x - \frac{b_2'}{b_1'} X_2 - \ldots \frac{b_n'}{b_n'} X_n \qquad \text{(Eq. B-26)}$$

These equations are the regressions of "observed" values on "observed" values, with the first used to predict ln y and the second or third to predict ln x. Because of the errors in both ln x and ln y, the regression of "true" values on "true" values will generally be steeper than given by the first two equations (5), and the "true" value of b_1 and $\frac{1}{b_1'}$. Under these conditions, Davies (5) suggests that it may be desirable to assume a "functional" relationship between y and x and he gives a technique for calculating it.

The slope S of the "functional" relationship is given by:

$$S = m + (m^2 + k^2)^{\frac{1}{2}} \qquad \text{(Eq. B-27)}$$

where

$$k^2 = \sigma^2_{\ln y} / \sigma^2_{\ln x}$$

and

$$m = \frac{1}{2b_1'} - \frac{k^2}{2b_1} \qquad \text{(Eq. B-28)}$$

$\sigma^2_{\ln y}$ and $\sigma^2_{\ln x}$ are respectively the variances of the dependent and independent variables. They can be derived from regression analyses using individual instrument readings in one type as the dependent variable and the mean of the type as the independent variable. b_1 and b_1' are given by equations B-24 and B-25.

The value of the slope S (5) should lie between those of b_1 and $\frac{1}{b_1'}$. Having obtained the slope of the "functional" relationship one can use the equation:

$$\ln\left(\frac{y}{\bar{x}^S}\right) = b_0'' + b_2''X_2 + \ldots + b_n''X_n \qquad \text{(Eq. B-29)}$$

where

$\ln\left(\frac{y}{\bar{x}^S}\right) = \ln y - S \ln \bar{x}$ is the dependent variable,

and $\bar{x}$ is the mean of the concentrations estimated by the reference instruments in the one dust cloud.

In applying this technique it was found difficult to obtain sufficiently accurate values for k^2 and it seems justified to assume a linear relationship between the two instruments being compared when b_1 and $\frac{1}{b_1'}$ bracket the value 1 as occurred in all the comparisons except those with the konimeters. Thus, in most cases the equation above was solved with the slope S set equal to 1.

Another examination of the konimeter concentrations plotted against those given by other instruments suggests that the four konimeters differ appreciably in behaviour and that there is no simple way of expressing the functional relationship. In view of this, the comparisons between the konimeters and the other instruments were estimated, not rigorously, by using the equation:

$$\ln\left(K^{s}/\bar{x}\right) = b_0''' + b_2'''X_2 + \ldots + b_n'''X_n \qquad \text{(Eq. B-30)}$$

where K is the dust concentration estimated by the konimeter, $\bar{x}$ is that estimated by the other instruments, and s is given by:

$$s = \frac{1}{S} = \frac{\frac{1}{b_1} + b_1'}{2} \qquad \text{(Eq. B-31)}$$

where b_1 and b_1' are determined by the Equations B24 and B25 with ln K as the dependent variable and independent variable respectively.

CONCLUSIONS

The independent variables of Equation B-29 meet the assumptions about errors and the regression analysis can be assumed to be applicable. This equation assumes a "functional" relationship between the estimates of dust concentration by the test and reference instruments in a given dust cloud of the form:

$$y = Ax^{S}$$

where A and S are constants.

The values of A will be different in each type of dust cloud and are obtained by the multiple linear regression analysis using Equation B 29. The value of S is assumed to be the same in each type of dust cloud and can be obtained from Equations B-24, B-25, and B-31. In many comparisons, S is near unity and a linear relationship was assumed.

ACKNOWLEDGEMENTS

The authors wish to thank Messrs. L. Casey and G. S. Rajhans of the Mining Research Centre for their assistance in setting up this program. They also wish to thank Mrs. Taylor, Messrs. Sumner and Evans of the Department's computer centre for their advice and assistance; also

Dr. Gray for his helpful comments and criticism.

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APPENDIX C
COMPARISON OF DUST SAMPLING INSTRUMENTS: TABLES OF RESULTS

The Tables C-1 to C-26 give full results of all the tests made in each type of dust cloud. Tables C-1 to C-20 are the main experiment. C-21 and C-22 are in dust clouds of aggregated particles. Tables C-23 to C-25 give the results at low concentrations obtained after fitting a high efficiency filter to the air intake on the chamber. C-26 gives the results of comparison of microscope techniques.

FOOTNOTES TO TABLES C-1 TO C-26

The flow rates for the various instruments are given below:

Midget Impinger	2.8 litres/minute (0.1 CFM)
Gathercole	5 cm^3 Snap Sample
Haslam	" "
Sartorius I	" "
Sartorius II	2.5 cm^3 Snap Sample
STP	7 cm^3/minute
LPDS Head	6 cm^3/minute
LPDS	2 cm^3/minute
Hexhlett	50 litres/minute
Electrostatic	85 litres/minute
High Volume	102 litres/minute
Medium Volume	20 litres/minute

Footnotes:

1. The counts given are for the konimeter samples prior to heat treatment and acid wash.
2. Standard Thermal Precipitator (STP).
3. Long-period dust sampler head without respirable dust size selector (LPDS).
4. Too high to count (THTC).
5. Too low to measure (TLTM).
6. Total count single particles and aggregates.
7. Aggregates per cubic centimetre (a/cm^3).

TABLE C-1
Concentration Measurements
Material: Coal: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm^3/hour					
	1.025	1.025	2.88	2.88	6.6	6.6
	Dust Concentration by Number; p/cm^3 ($> 5\ \mu m$)					
Midget Imp.	0	0	2.5	1.27	7.66	0
Midget Imp.	0.6	0.65	2.5	0	0	0
Konimeters [1]						
Gathercole	13.00	21.2	7.7	16.24	20	
Haslam	14.1	12.1	31.2	33.8	104	6.98
Sartorius	7.9	6.4	16.1	12.2	22.6	16.1
Sartorius	11.0	11.6	12.9	9.3	14	10.4
STP [2]	0	0	0	0	0	0
LPDS [3] Head	0	0	0	3.3	2	2
Midget Imp.	0	0	0	0	0	0
LPDS	1.6	1.6	0	0	0	1
	Dust Concentration by Number; p/cm^3 ($< 5\ \mu m$)					
Midget Imp.	139	142	226	244	495	560
Midget Imp.	122	132	221	246	567	546
Konimeters						
Gathercole	1157	1263	2438	2174	6042	THTC [4]
Haslam	882	858	1304	1326	2492	2392
Sartorius	1132	1268	2364	2160	4664	4092
Sartorius	1291	1471	3064	3064	6028	5436
Midget Imp.	117.5	136	200	232	585	518
	Dust Concentration by Number; p/cm^3 ($1-5\ \mu m$)					
STP	195	247	407	360	712	666
LPDS Head	192	187	475	465	1103	991
LPDS	273	250	477	425	1649	936
	Dust Concentration by Number; p/cm^3 ($1/2-5\ \mu m$)					
STP	730	752	1517	1247	2327	2476
LPDS Head	485	519	1525	1430	3635	3911
LPDS	724	638	1537	1415	5307	3608
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	5.45	5.75	6.60	6.95	11.7	11.85
	Dust Concentration by Mass; mg/m^3					
Hexhlett	0.74	0.85	1.38	1.64	3.48	3.78
NCB Grav.	0.55	0.65	1.2	1.8	2.6	2.8
Electrostatic	0.92	1.16	1.53	1.78	4.76	3.65
High Volume	0.86	0.94	1.70	1.47	4.05	4.2
Medium Volume	0.79	1.03	1.56	1.27	4.02	3.84

Footnotes on page -2

TABLE C-2
Concentration Measurements
Material: Coal: Dispersion Method: Pulverized

Sampler	Dust Feed Rate cm^3/hour					
	6.6	20	20.5	57	57.5	114
	Dust Concentration by Number; p/cm^3 (> 5 μm)					
Midget Imp.	10	10	13	14	47	16
Midget Imp.	5.8	14.5	25	14	39	32
Konimeters [1]						
Gathercole	6.4	.45	17		15.4	
Haslam	7.7	1.2	52.8	1.0	74.8	
Sartorius	3.2	2.5	11.6		11.8	
Sartorius	4.3	5.0	16.7	1.8	16	2.3
STP [2]	3.4	9		17	10	32
LPDS [3] Head	3.2	19	3	24	20	200
Midget Imp.	2.5	1.2	6.5	4	2.6	21
LPDS	0	3	9		18	81
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	121	118	490	427	635	660
Midget Imp.	118	155	485	477	735	618
Konimeters						
Gathercole	1400	1041	3308	THTC [4]	4182	THTC
Haslam	800	448	2258		2959	THTC
Sartorius	1556	1138	4106		5231	THTC
Sartorius	1734	1374	4878	2242	7012	3650
Midget Imp.	116	129	450	437	805	650
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	244	270		1045	1245	2000
LPDS Head	230	384	782	980	1730	3160
LPDS	173	530	984		1530	2260
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	797	1010		2417	3665	3900
LPDS Head	943	1184	2422	2190	3365	5030
LPDS	593	1380	3522		4160	3460
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	4.37	5.7	8.9	6.8	10.6	10.0
	Dust Concentration by Mass; mg/m^3					
Hexhlett	0.88	1.43	4.05	3.9	4.90	6.87
NCB Grav.	0.8	1.85	4.8	6.0	3.6	9.0
Electrostatic	1.39	2.17	5.86	6.62	7.95	10.1
High Volume	1.63	2.47	5.80	5.22	8.86	10.7
Medium Volume	1.48	3.0	5.74	7.32	7.85	11.4

Footnotes on page C-2

TABLE C-3
Concentration Measurements
Material: Coal: Dispersion Method: Ejector - Cyclone

Sampler	Dust Feed Rate cm³/hour					
	6.6	20	20.5	57	57.5	114
	Dust Concentration by Number; p/cm³ (> 5 μm)					
Midget Imp.	7	14	17	63	55	196
Midget Imp.	8.5	15	14	64	65	150
Konimeters [1]						
Gathercole	5.6	1.6	13.8		59.4	
Haslam	94.6	2.1	46	5.0	79.1	
Sartorius	6.9	1.25	10.7	3.0	46.2	
Sartorius	9.2	4.7	13.2	5.0	80.3	
STP [2]	8.3	18	12	12	45.5	79
LPDS [3] Head	13.5	34	13	87	60	141
Midget Imp.	2.5	8	10	14	21	75
LPDS	3		3	47	25	56
	Dust Concentration by Number; p/cm³ (< 5 μm)					
Midget Imp.	91	178	192	500	485	1100
Midget Imp.	79.5	180	192	460	487	895
Konimeters						
Gathercole	422	859	876	THTC	2072	THTC
Haslam	1183	531	644	1149	1430	THTC
Sartorius	612	954	922	1738	2430	THTC
Sartorius	859	1218	1089	2113	2920	THTC
Midget Imp.	77	175	168	585	449	865
	Dust Concentration by Number; p/cm³ (1-5 μm)					
STP	143	425	256	1499	695	1670
LPDS Head	116	556	222	1610	785	1900
LPDS	126		330	1430	925	2260
	Dust Concentration by Number; p/cm³ (1/2-5 μm)					
STP	277	830	503	2386	1337	3420
LPDS Head	197	1112	415	2385	1273	3565
LPDS	224		532	2360	1565	4480
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	4.05	4.5	3.70	8.0	6.30	10.7
	Dust Concentration by Mass; mg/m³					
Hexhlett	1.44	2.56	1.79	6.45	5.45	14.70
NCB Grav.	1.1	3.50	1.5	8.0	5.6	15.60
Electrostatic	1.56	4.42	3.12	11.4	10.30	27.2
High Volume	1.26	5.05	3.46	11.3	12.10	28.0
Medium Volume	1.59	4.4	3.08	11.1	10.79	29.8

Footnotes on page G-2

TABLE C-4
Concentration Measurements
Material: Coal: Dispersion Method: Ejector - No Cyclone

Sampler	Dust Feed Rate cm³/hour					
	6.6	20	20.5	57	57.5	114
	Dust Concentration by Number; p/cm³ (> 5 μm)					
Midget Imp.	10	46	22	80	78	109
Midget Imp.	8	46	28.4	62	73	164
Konimeters[1]						
Gathercole	7	0.8	31.3		60.4	
Haslam	14.6	1.6	38.3	8.5	10.4	
Sartorius	8.4	1.2	9.8		50.1	
Sartorius	15.4	1.6	14.6	7	70.8	
STP[2]	3.2	16	12	28	36	114
LPDS[3] Head	10.5	24	21	130	48	220
Midget Imp.	2	34	16	31	15	49
LPDS	2.3		25	4	37	131
	Dust Concentration by Number; p/cm³ (< 5 μm)					
Midget Imp.	98	203	246	380	510	980
Midget Imp.	92	145	206	475	425	1080
Konimeters						
Gathercole	508	794	1222	THTC[4]	1029	THTC
Haslam	292	461	532	1154	344	THTC
Sartorius	483	775	1010	THTC	2179	THTC
Sartorius	662	1014	1246	2324	2806	THTC
Midget Imp.	80	146	220	375	435	950
	Dust Concentration by Number; p/cm³ (1-5 μm)					
STP	99.5	325	223	965	658	1780
LPDS Head	97.5	470	355	1530	800	2280
LPDS	107		342		780	2500
	Dust Concentration by Number; p/cm³ (1/2-5 μm)					
STP	184.5	647	410	1445	1210	3710
LPDS Head	172.5	965	584	2435	1423	5020
LPDS	205		500		1357	4540
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	4.22	4.1	5.35	8.2	6.80	9.8
	Dust Concentration by Mass; mg/m³					
Hexhlett	0.95	2.28	2.48	7.38	5.41	14.75
NCB Grav.	0.95	2.70	2.5	8.70	5.0	17.2
Electrostatic	2.77	5.59	5.29	18.5	15.1	40.0
High Volume	1.74	6.55	6.25	19.3	17.2	42.7
Medium Volume	2.36	6.42	5.95	20.8	14.3	45.0

Footnotes on page C-2

TABLE C-5
Concentration Measurements
Material: Silica: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm^3/hour					
	1.025	1.025	2.68	2.88	6.6	6.6
	Dust Concentration by Number; p/cm^3 (> 5 μm)					
Midget Imp.	0.65	0.65	2.59	16.6	36	31
Midget Imp.	0.65	3.9	1.29	10.1	46.7	44
Konimeters [1]						
Gathercole	1.8	1.04	4.52		22.04	22.68
Haslam	7.4	3.2	17.8	16.3		
Sartorius	2.4	1.52	2.96	3.96	17.80	17.8
Sartorius	3.84	1.60	5.92	6.32	31.2	35.8
STP [2]	5	3.35	4	11.7	57	48
LPDS [3] Head	0	3.0	3	24	69	51
Midget Imp.	0	0	0	0	0	5.17
LPDS	4.6	0	0	9.3	0	0
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	206	167	482	324	1206	1287
Midget Imp.	191	155	426	396	1285	1164
Konimeters						
Gathercole	933	1030	1844	THTC [4]	3234	3036
Haslam	1012	908	2084	2366	THTC	THTC
Sartorius	1292	1508	2536	2134	3971	2703
Sartorius	1885	1782	3517	2842	5608	4075
Midget Imp.	196	160	384	342	1368	1020
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	264	252	572	452	1197	830
LPDS Head	378	332	829	585	1452	1324
LPDS	479	400	855	1078	1413	1302
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	919	960	1732	1512	3457	2520
LPDS Head	1375	1310	2948	2045	3592	3980
LPDS	1437	1302	2724	2928	4110	3701
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	9.70	8.90	11.20	11.8	16.00	15.70
	Dust Concentration by Mass; mg/m^3					
Hexhlett	1.94	1.67	3.72	3.78	11.65	9.86
NCB Grav.	1.64	1.20	4.6	4.7	13.0	10.60
Electrostatic	2.26	1.84	4.54	5.65	17.65	17.55
High Volume	2.22	2.30	5.40	5.98	21.50	19.40
Medium Volume	2.30	2.14	4.86	5.75	19.30	17.82

Footnotes on page C-2

TABLE C-6
Concentration Measurements
Material: Silica: Dispersion Method: Pulverised

Sampler	Dust Feed Rate cm³/hour					
	6.6	6.6	16.1	16.1	59.4	59.4
	Dust Concentration by Number; p/cm³ (> 5 µm)					
Midget Imp.	6.5	8.4	20.7	9.1	57	67.2
Midget Imp.	7.8	3.9	18.1	11.7	75	36.2
Konimeters [1]						
Gathercole	4.1	4.1	8.5	7.3	16.9	14
Haslam	16.5	10	13.4		46.5	45.4
Sartorius	2.4	4.6	10		11.9	9.2
Sartorius	4.3	6.5	10.6		18.9	12.5
STP [2]	4.5	7	7.5	9	38	38
LPDS [3] Head	10.4	9	18.5	19	61	29
Midget Imp.	1.3	0.65	1.3	0	5.2	13
LPDS	3.3	0	3	0	25	12
	Dust Concentration by Number; p/cm³ (< 5 µm)					
Midget Imp.	153	168	318	244	619	866
Midget Imp.	165	188	268	312	835	880
Konimeters						
Gathercole	872	900	1304	1329	2204	1956
Haslam	1039	779	1165		2352	2362
Sartorius	921	844	1026		2664	2170
Sartorius	1160	1111	1475		3211	2802
Midget Imp.	165	209	252	264	754	825
	Dust Concentration by Number; p/cm³ (1-5 µm)					
STP	176	196	296	296	912	951
LPDS Head	224	204	380	508	1200	945
LPDS	230	206	418	408	1045	1010
	Dust Concentration by Number; p/cm³ (1/2-5 µm)					
STP	604	633	959	986	2602	2631
LPDS Head	768	690	1165	1693	2945	2915
LPDS	695	649	1283	1156	2905	3230
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	4.15	4.08	5.53	5.44	8.35	8.45
	Dust Concentration by Mass; mg/m³					
Hexhlett	1.14	1.25	2.13	2.01	6.73	6.16
NCB Grav.	1.75	1.50	3.5	2.2	6.0	4.8
Electrostatic	2.22	2.41	4.21	6.03	13.0	12.9
High Volume	2.74	3.40	5.35	5.20	17.8	15.8
Medium Volume	2.67	2.89	4.65	4.50	15.5	13.9

Footnotes on page C-2

TABLE C-7
Concentration Measurements
Material: Silica: Dispersion Method: Ejector - Cyclone

Sampler	Dust Feed Rate cm^3/hour					
	6.6	11.3	20.5	34	57.5	102
	Dust Concentration by Number; p/cm^3 ($> 5\ \mu m$)					
Midget Imp.	3.9	2	27.2		28.5	24
Midget Imp.	1.9	3	11.7	19	20.8	55
Konimeters [1]						
Gathercole	1.45		8.2		19	
Haslam	5.85		10.5		37.4	
Sartorius	3.1		3.6		7.3	
Sartorius	3.6		7.0		10.6	
STP [2]	2.6	5	5	6	25.8	14
LPDS [3] Head	3.8	5	11.3	35	40	130
Midget Imp.	0.65	0	1.3	0	2.6	5
LPDS	5.8	4	6.3	15	12.7	21
	Dust Concentration by Number; p/cm^3 ($< 5\ \mu m$)					
Midget Imp.	100	180	333	540	610	1140
Midget Imp.	95.5	220	360	545	625	1120
Konimeters						
Gathercole	368	710	1124	1210	1928	THTC [4]
Haslam	376	520	1204		2556	THTC
Sartorius	277	855	1300		2292	3030
Sartorius	349	875	1856		3154	3309
Midget Imp.	71.9	228	250	465	540	1070
	Dust Concentration by Number; p/cm^3 ($1-5\ \mu m$)					
STP	64.2	420	264	740	595	960
LPDS Head	88	340	332	845	825	2380
LPDS	110	405	255	975	600	1660
	Dust Concentration by Number; p/cm^3 ($1/2-5\ \mu m$)					
STP	172.2	765	749	1390	1600	1560
LPDS Head	173	585	777	1725	1825	3960
LPDS	261	690	810	1875	1436	3220
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	3.88	6.5	5.28	9.0	7.65	13.5
	Dust Concentration by Mass; mg/m^3					
Hexhlett	0.56	1.4	2.47	4.0	5.23	9.2
NCB Grav.	0.7		1.6		5.0	
Electrostatic	0.93	2.2	4.75	6.6	10.85	16.4
High Volume	1.17	2.2	5.66	6.3	10.7	15.5
Medium Volume	1.05		4.57		9.86	

Footnotes on page C-2

TABLE C-8
Concentration Measurements
Material: Silica: Dispersion Method: Ejector - No Cyclone

Sampler	Dust Feed Rate cm³/hour					
	6.6	11.3	20.5	34	57.5	102
	Dust Concentration by Number; p/cm³ (> 5 µm)					
Midget Imp.	5.2	11	16.8	21	46	
Midget Imp.	3.2	16	22	20	67.5	50
Konimeters [1]						
Gathercole	3.6		17.3		15.1	
Haslam	8.0		17.4		40.2	
Sartorius	2.9		5.5		17.7	
Sartorius	3.8		6.5		22.6	
STP [2]	3.2	1	8.1	21	17.2	4
LPDS [3] Head.	6.7	21	30	50	22	42
Midget Imp.	1.3	0	2.6	0	5.2	0
LPDS	0	12	16	12	12.4	4
	Dust Concentration by Number; p/cm³ (< 5 µm)					
Midget Imp.	86.2	206	318	585	575	1760
Midget Imp.	93.5	240	332	550	615	1650
Konimeters						
Gathercole	528	1410	1308	1695	1726	THTC [4]
Haslam	470		1360	1180	2164	THTC
Sartorius	340	1425	1486	1955	1913	3215
Sartorius	520	1364	2015	1774	2353	2845
Midget Imp.	87	195	140	585	580	1000
	Dust Concentration by Number; p/cm³ (1-5 µm)					
STP	49.5	190	258	1065	710	2650
LPDS Head	98	230	540	760	570	1770
LPDS	129	370	530	855	713	1220
	Dust Concentration by Number; p/cm³ (1/2-5 µm)					
STP	148	690	668	1955	1795	3300
LPDS Head	221	445	1180	1330	1435	3030
LPDS	295	665	1085	1735	1698	3020
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope.	4.50	6.5	6.98	7.8	8.90	13.0
	Dust Concentration by Mass; mg/m³					
Hexhlett	0.74	1.8	2.83	4.4	5.49	10.0
NCB Grav.	0.7		3.0		6.0	
Electrostatic	1.88	3.5	7.45	12.2	14.8	27.6
High Volume	2.40	3.4	9.9	10.6	18.6	23.6
Medium Volume	2.08		18.6		16.0	

Footnotes on page C-2

TABLE C-9
Concentration Measurements
Material: Pyrite: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm³/hour					
	0.25	0.25	0.51	0.51	1.025	1.025
	Dust Concentration by Number; p/cm³ (> 5 μm)					
Midget Imp.	0	1	0	2	3	0
Midget Imp.	6	3	0	0	0	0
Konimeters [1]						
Gathercole	1.12	1.4			9.24	
Haslam	3.1	2.3	3.5	12.3		
Sartorius	0.96	1.6	2.2	1.8		
Sartorius	0.96	1.4	1.4	2.4	2.3	4.0
STP [2]	0	0	0	0	0	4.5
LPDS [3] Head	0	0	0	3	0	6.5
Midget Imp.	0	0	0	0	0	0
LPDS	0	0	0	0	0	0
	Dust Concentration by Number; p/cm³ (< 5 μm)					
Midget Imp.	435	407	869	1000	1902	1820
Midget Imp.	405	378	853	903	2001	1620
Konimeters						
Gathercole	2562	2322	THTC [4]	THTC	2137	THTC
Haslam	1894	1948	2736	3504	THTC	THTC
Sartorius	2274	2744	3590	2990		
Sartorius	3088	3722	4640	3884	6764	6136
Midget Imp.	430	381	870	762	1872	1910
	Dust Concentration by Number; p/cm³ (1-5 μm)					
STP	207	244	505	483	1300	1640
LPDS Head	156	153	440	435	1720	1098
LPDS	242	358	344	735	1520	1730
	Dust Concentration by Number; p/cm³ (1/2-5 μm)					
STP	1957	1834	3465	3605	7800	7840
LPDS Head	2047	1681	3820	4327	9460	8248
LPDS	2635	2845	4688	5171	9620	10080
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	5.45	5.45	8.58	8.40	11.72	11.4
	Dust Concentration by Mass; mg/m³					
Hexhlett	1.50	1.50	3.005	2.85	6.51	5.45
NCB Grav.	2.35	1.55	5.0		6.2	4.0
Electrostatic	1.92	1.98	3.38	3.40	9.96	6.1
High Volume	1.85	2.06	4.05	4.08	8.33	7.14
Medium Volume	1.29	1.92	4.45	3.56	8.01	6.95

Footnotes on page C-2

TABLE C-10
Concentration Measurements
Material: Pyrite: Dispersion Method: Pulverised

Sampler	Dust Feed Rate cm^3/hour					
	6.6	6.6	20.5	20.5	57.5	57.5
	Dust Concentration by Number; p/cm^3 (> 5 μm)					
Midget Imp.	2.5	6.1	14.4	14.3	31	23
Midget Imp.	1	1.9	13	19.5	36	28
Konimeters[1]						
Gathercole	1.3	1.3	2.5	3.6		
Haslam	6.5	8.5	7.2	16.5		
Sartorius	3.5	2.1	5.7	3.1		
Sartorius	10.0	4.0	14.5	4.7	7	13
STP[2]	2.7	4.7	5.1	5.2	25.2	16.6
LPDS[3] Head	2.8	5.7	22.4	24	25	30
Midget Imp.	1.6	0.65	2.6	1.3	10	5.2
LPDS	0	0	9.2	12.3	0	12.8
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	101	210	332	481	810	995
Midget Imp.	117	100	375	522	825	900
Konimeters						
Gathercole	627	536	1352	1342		
Haslam	1016	747	1606	2214		
Sartorius	705	584	1154	1073		
Sartorius	748	683	1758	1312	2215	2754
Midget Imp.	193	116	320	403	770	879
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	133	108	340	290	635	765
LPDS Head	145	138	525	482	1220	1240
LPDS	102	148	485	510	790	1290
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	1058	1108	2380	2100	4245	4460
LPDS Head	995	1258	3295	3342	7320	6040
LPDS	968	1108	2585	3220	5090	5590
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	6.32	6.64	7.60	7.96	10.40	10.42
	Dust Concentration by Mass; mg/m^3					
Hexhlett	1.08	1.13	2.83	2.91	5.69	5.8
NCB Grav.	1.5	1.4	3.7	3.6	6.4	6.4
Electrostatic	1.57	1.96	4.8	4.95	10.3	10.2
High Volume	2.18	2.39	5.15	5.35	11.1	11.3
Medium Volume	2.01	2.00	5.65	5.65	11.2	11.7

Footnotes on page C-2

TABLE C-11
Concentration Measurements
Material: Pyrite: Dispersion Method: Ejector - Cyclone

Sampler	Dust Feed Rate cm³/hour					
	6.6	11.3	20.5	34	57.5	102
	Dust Concentration by Number; p/cm³ (> 5 μm)					
Midget Imp.	3.9	1	14.5	3.5	39	15
Midget Imp.	5.8	1	27.1	6.5	54.5	28
Konimeters [1]						
Gathercole	3.84		8.5			
Haslam	7.7		21.8			
Sartorius	1.6		6.6			
Sartorius	3.0		12.7		12.2	
STP [2]	1.5	2	13	18	17	19
LPDS [3] Head	3.9	4	6.3	8	36	29
Midget Imp.	0	0	5.2	0	10.8	0
LPDS	1.5	0	6.2	15	0	0
	Dust Concentration by Number; p/cm³ (< 5 μm)					
Midget Imp.	117	47	318	167	585	304
Midget Imp.	110	45	384	171	705	392
Konimeters						
Gathercole	400	869	1124	1232		THTC [4]
Haslam	533	619	1036	1192		THTC
Sartorius	280	852	1086	1480		THTC
Sartorius	337	1114	1132	1968	2667	2959
Midget Imp.	93.5	43	266	172	630	363
	Dust Concentration by Number; p/cm³ (1-5 μm)					
STP	57	219	246	810	540	710
LPDS Head	59	500	313	915	706	1765
LPDS	73	315	196	1180	770	1480
	Dust Concentration by Number; p/cm³ (1/2-5 μm)					
STP	161	437	628	1610	1378	1075
LPDS Head	148	846	738	1398	1396	2602
LPDS	210	579	616	1850	1680	2645
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	3.39	5.0	4.92	6.3	5.75	8.0
	Dust Concentration by Mass; mg/m³					
Hexhlett	0.75	1.8	2.43	3.9	6.31	9.7
NCB Grav.	0.65		2.9		7.0	
Electrostatic	1.87	3.2	6.10	8.7	18.1	20.3
High Volume	2.30	3.4	6.47	9.0	21.6	
Medium Volume	1.68		5.65		17.5	

Footnotes on page C-2

TABLE C-12
Concentration Measurements
Material: Pyrite: Dispersion Method: Ejector - No Cyclone

Sampler	Dust Feed Rate cm^3/hour					
	6.6	11.3	20.5	34	57.5	102
	Dust Concentration by Number; p/cm^3 (> 5 μm)					
Midget Imp.	4.5	2.7	10.8	8	67	15
Midget Imp.	4.5	4.6	26	12	78	24
Konimeters [1]						
Gathercole	3.9		8.5			
Haslam	8.3		33			
Sartorius	4.4		10.2			
Sartorius	9.8		13.7		26.6	
STP [2]	4.8	7	10.9	33		51
LPDS [3] Head	5.6	46	43		114	150
Midget Imp.	0	0.4	1.3	0	5.2	0
LPDS	0	5	6.1	0	0	0
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	110	85	337	173	976	320
Midget Imp.	156	66	431	222	1135	400
Konimeters						
Gathercole	500	1009	1364	THTC [4]		THTC
Haslam	367	575	909	1344		THTC
Sartorius	414		936	THTC		THTC
Sartorius	615		1682	3393	2829	THTC
Midget Imp.	114	85	338	189	1080	364
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	74	360	196	945		2490
LPDS Head	81	475	315		706	2310
LPDS	78	415	272	1040	606	2300
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	208	571	578	1535		3990
LPDS Head	201	724	728		1581	3750
LPDS	255	673	797	1450	1756	3600
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	3.46	5.2	4.13	6.0	8.55	9.2
	Dust Concentration by Mass; mg/m^3					
Hexhlett	0.975	2.05	2.9	4.9	7.43	12.4
NCB Grav.	1.5		3.5		7.8	
Electrostatic	4.00	8.1	12.8	20	30.0	51
High Volume	4.88	8.1	14.9	21	40.6	54
Medium Volume	4.10		14.0		33.2	

Footnotes on page C-2

TABLE C-13
Concentration Measurement
Material: Mica: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm^3/hour					
	2.88	2.88	6.6	6.6	16.1	16.1
	Dust Concentration by Number; p/cm^3 (> 5 μm)					
Midget Imp.	8	7	39	42.7	112	82
Midget Imp.	11	13	21	31	82	93
Konimeters [1]						
Gathercole	23.9	23.7	58.6	39.2		50.2
Haslam	13.8	13.3	25.8	32.5		87.7
Sartorius	13.9	17.1	16.7	20.3		
Sartorius	23.2	16.4	26.4	22.5		
STP [2]	2	7	15	24.5	169	57
LPDS [3] Head	1.5	6	41	24	103	79
Midget Imp.	9	11	25	17	62	58
LPDS	6	9	55	0	56	0
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	475	470	883	712	2550	1850
Midget Imp.	413	492	940	667	2030	2015
Konimeters						
Gathercole	1404	1830	2723	2899	THTC [4]	THTC
Haslam	1891	1871	3327	2838	THTC	THTC
Sartorius	2019	1866	2558	2091	THTC	3596
Sartorius	2742	2448	3477	2843	THTC	5145
Midget Imp.	444	458	746	775	2500	1830
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	512	245	1040	800	2860	1950
LPDS Head	436	355	1035	735	2230	2285
LPDS	535	567	1085	1015	3070	3345
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	1577	638	2910	2430	6600	5320
LPDS Head	1249	1139	2955	2175	6130	6155
LPDS	1555	2057	2925	2700	7610	8325
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	8.50	9.30	13.9	12.6	24.0	20.5
	Dust Concentration by Mass; mg/m^3					
Hexhlett	2.775	3.00	7.375	6.90	23.5	18.99
NCB Grav.	2.65	2.75	7.2	6.2	21.0	16.6
Electrostatic	2.98	3.20	7.75	7.80	26.5	24.2
High Volume	3.30	3.76	8.15	7.80	27.6	23.4
Medium Volume	3.16	3.50	8.50	8.25	28.3	24.1

Footnotes on page C-2

TABLE C-14
Concentration Measurements
Material: Mica: Dispersion Method: Pulverization

Sampler	Dust Feed Rate cm^3/hour 5	7	7.5	16.1	17	59
	Dust Concentration by Number; p/cm^3 (> 5 μm)					
Midget Imp.	8	2	10	34	8	26
Midget Imp.	7	1	10	34	12	39
Konimeters [1]						
Gathercole	5.4		5.4	20		
Haslam	16		17.5	33.3		
Sartorius	7.9		8.8	11.6		
Sartorius	13.2		19.6	17		
STP [2]	7	1	14	11.5	0	44
LPDS [3] Head	13	0	15	32	7	64
Midget Imp.	4	3	5	10	1	15
LPDS	0	1	9	18	15	31
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	228	249	306	566	760	1790
Midget Imp.	228	226	366	638	699	1990
Konimeters						
Gathercole	1092	985	928	1487		
Haslam	1616		1571	2393		
Sartorius	1997	1586	1786	2428		
Sartorius	3068	2193	2684	3865	THTC [4]	THTC
Midget Imp.	224	230	330	537	749	1960
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	139	472	236	1025	1020	2580
LPDS Head	187	480	269	610	1325	2460
LPDS	95	705	305	625	1090	2450
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	648	1560	976	3165	3240	8100
LPDS Head	717	1500	1214	2060	4210	9750
LPDS	335	1960	1259	1950	4960	9530
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	7.47	7.9	8.82	9.97	13.8	24.3
	Dust Concentration by Mass; mg/m^3					
Hexhlett	1.88	2.26	2.46	3.09	6.42	20.22
NCB Grav.	1.45	3.05	1.9	3.6	7	20
Electrostatic	2.33	2.32	3.30	7.15	7	22.6
High Volume	2.80	3.13	3.87	7.75	8.02	25.05
Medium Volume	2.54	2.97	3.60	9.80	8.38	28.65

Footnotes on page C-2

TABLE C-15
Concentration Measurements
Material: Mica: Dispersion Method: Ejector - Cyclone

Sampler	Dust Feed Rate cm³/hour					
	2.88	6.6	7	16.1	17	59
	Dust Concentration by Number; p/cm³ (> 5 μm)					
Midget Imp.	10	11.5	19	34	58	40
Midget Imp.	14	18	14	26	42	38
Konimeters [1]						
Gathercole	10.44	20.8		33.3		
Haslam	36.8	46.8				
Sartorius	4.8	7.9				
Sartorius	8.6	13.7		48.6		
STP [2]	14.5	26	17	57	62	198
LPDS [3] Head	7.3	26	21	57	42	96
Midget Imp.	2.5	5	8	15.5	8	14
LPDS	1.5	6.2	14	12.5	9	55
	Dust Concentration by Number; p/cm³ (< 5 μm)					
Midget Imp.	330	617	494	1400	1285	3800
Midget Imp.	320	600	530	1600	1370	3780
Konimeters						
Gathercole	1016	1573	1723	3057	THTC [4]	
Haslam	1914	2225	1350	THTC	THTC	
Sartorius	1515	2336	THTC		THTC	
Sartorius	1413	2890	2553	6455	3907	THTC
Midget Imp.	314	584	500	1550	1380	3580
	Dust Concentration by Number; p/cm³ (1-5 μm)					
STP	312	588	480	1225	1055	3580
LPDS Head	275	545	430	1290	715	3900
LPDS	309	620	590	1625	1290	3900
	Dust Concentration by Number; p/cm³ (1/2-5 μm)					
STP	765	1318	1280	3095	3300	10800
LPDS Head	568	1285	1640	3080	2820	10700
LPDS	679	1343	1790	3640	4050	10710
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	6.97	9.4	11.8	15.7	15.8	27.3
	Dust Concentration by Mass; mg/m³					
Hexhlett	2.56	4.25	2.67	10.4	12.3	35.95
NCB Grav.	2.6	4.3	2.75	10.4	13.50	41
Electrostatic	3.26	5.64	5	14.7	14.4	48.5
High Volume	3.78	6.95	6.72	16.0	15.47	45.75
Medium Volume	3.66	6.16	7.12	15.3	17.85	55.76

Footnotes on page C-2

TABLE C-16
Concentration Measurements
Material: Mica: Dispersion Method: Ejector - No Cyclone

Sampler	Dust Feed Rate cm^3/hour					
	2.88	6.6	7	16.1	17	59
	Dust Concentration by Number; p/cm^3 ($> 5\ \mu m$)					
Midget Imp.	14	13	22	75	30	41
Midget Imp.	12	15.5	18	57	33	49
Konimeters [1]						
Gathercole	8.1	14.7		43.2		
Haslam	36.3	52.4				
Sartorius	5.9	16.4				
Sartorius	9.4	28.2		35.1		
STP [2]	9	24	16	118	70	166
LPDS [3] Head	12.5	13	21	90	92	152
Midget Imp.	6	2.5	6	26	0	0
LPDS	8	6	5	0	9	183
	Dust Concentration by Number; p/cm^4 ($< 5\ \mu m$)					
Midget Imp.	260	506	538	1220	1220	3460
Midget Imp.	260	540	528	1440	1310	3480
Konimeters						
Gathercole	1428	1538	1842	3102	THTC [4]	
Haslam	1336	2206	1407	THTC	THTC	
Sartorius	1368	2000	1451		THTC	
Sartorius	1702	2932	2082	5278	4096	THTC
Midget Imp.	276	491	513	1420	1200	3475
	Dust Concentration by Number; p/cm^3 ($1-5\ \mu m$)					
STP	265	415	380	1240	1400	3460
LPDS Head	275	431	426	880	1170	4700
LPDS	329	440	558	945	1460	4350
	Dust Concentration by Number; p/cm^3 ($1/2-5\ \mu m$)					
STP	671	1201	1340	3150	3830	8850
LPDS Head	637	1096	1450.	2025	3880	8650
LPDS	721	1175	1640	2605	4050	10200
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	6.95	9.08	11.3	15.7	15.7	25.6
	Dust Concentration by Mass; mg/m^3					
Hexhlett	2.34	4.05	4.72	11.24	12.27	34.55
NCB Grav.	1.9	2.8	5.20	8.2	14.50	36.
Electrostatic	3.55	5.6	6.59	15.9	17.1	47.5
High Volume	3.74	7.0	7.33	19.4	18.50	47.56
Medium Volume	3.65	6.2	7.80	17.9	19.70	56.75

Footnotes on page C-2

TABLE C-17
Concentration Measurements
Material: Asbestos: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm³/hour					
	1.025		2.88		6.6	
	Non Fibrous Dust Concentration by No. p/cm³ (> 5 μm)					
Midget Imp.	0	1.95	1.2	3.9	5.2	5.17
Midget Imp.	0	.65	0	1.3	5.2	7.8
Konimeters [1]						
Gathercole	6.52	2.08	29	12	12.0	8.24
Haslam		1.12	1.3	2.48		
Sartorius	1.84	1.2	2.28	1.7	2.15	2.4
Sartorius	8.16	5.9	4.0	4.5	5.3	2.8
STP [2]	.64	.64	0	3.79	7.5	2.6
LPDS [3] Head	3.26	3.1	7.0	3.2	106	12.9
Midget Imp.	0	0	0	0	0	0
LPDS		1.54		3.1		0
	Dust Concentration by Number; p/cm³ (< 5 μm)					
Midget Imp.	92.8	87.6	218	206	360	466
Midget Imp.	98.6	98.4	165	222	392	389
Konimeters						
Gathercole	2102	1200	2144	1774	3064	4708
Haslam	THTC	1018	1247	1648	THTC	THTC
Sartorius	1070	878	1771	1574	3604	3477
Sartorius	2799	2468	2968	2665	5924	4444
Midget Imp.	65	81.7	229	168	293	290
	Dust Concentration by Number; p/cm³ (1-5 μm)					
STP	3.5	4.46	11.6	6.3	2.51	20.5
LPDS Head	13.1	9.3	21.0	16.3	31.2	12.9
LPDS		1.54		15.1		4.95
	Dust Concentration by Number; p/cm³ (1/2-5 μm)					
STP	104.5	124.46	856.6	179.3	333.1	430.6
LPDS Head	211.1	93.8	903	175.3	429.2	412.9
LPDS		77.0		207.1		504.95
	Fibrous Dust Concentration by Number; p/cm³					
Midget Imp.	22.8	30.3	62.1	53	143	111
Midget Imp.	20.8	23.4	49.2	50.4	111	83
Konimeters						
Gathercole	.68	.28	0	2.5	2	1.36
Haslam		.84	0	.76		
Sartorius	1.04	1.52	1	.7	12.05	13
Sartorius	13.96	8.3	2	2.0	12.0	4.8
STP	447	366	845	879	1820	1820
LPDS Head	700	620	882	770	1300	2040
Midget Imp.	2.47	18.2	51.8	38.8	67.5	67.5
LPDS		382		1120		2710
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	6.90		7.70		9.75	10.95
	Dust Concentration by Mass; mg/m³					
Hexhlett	.88	.66	1.87	1.50	2.94	3.21
NCB Grav.	.95	.60	1.7	1.4	3.00	3.81
Electrostatic	1.19	.91	2.21	2.28	5.75	7.05
High Volume	1.31	1.27	2.75	3.05	6.45	6.96
Medium Volume	1.51	.81	3.06	2.68	5.66	6.44

Footnotes on Page C-2

TABLE C-18
Concentration Measurements
Material: Asbestos: Dispersion Method: Pulverized

Sampler	Dust Feed Rate cm^3/hour					
	2.88		6.6	16.1		
	Non Fibrous Dust Concentration by No. p/cm^3 (> 5 μm)					
Midget Imp.	4	10	13	11	8	16
Midget Imp.	8	8	13	17	10	16
Konimeters [1]						
Gathercole						
Haslam						
Sartorius		3.64	2.44	5.6		
Sartorius		6.64	4.2	25.8		17.4
STP [2]	2	11	9	4	6	12
LPDS [3] Head	0	7.5	9	22	6	12
Midget Imp.	3	4.5	13	6.5	10	8
LPDS	4	10	11	10	19	0
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	212	210	356	465	910	950
Midget Imp.	212	298	420	537	897	1050
Konimeters						
Gathercole						
Haslam						
Sartorius		2254	2312	1432		
Sartorius		3180	3884	3861		4921
Midget Imp.	166	262	406	505	655	1030
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	59	146	164	270	412	320
LPDS Head	97	159	262	388	426	390
LPDS	108	91	264	225	277	166
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	540	870	1025	1500	1690	1650
LPDS Head	650	1290	1400	1900	1945	2210
LPDS	580	1120	1280	1430	1790	1250
	Fibrous Dust Concentration by Number; p/cm^3					
Midget Imp.	13	13	18	20	31	34
Midget Imp.	14	12	27	28	41	41.5
Konimeters						
Gathercole						
Haslam						
Sartorius		1.32	5.16	0.8		
Sartorius		1.28	8.2	5.1		4.6
STP	128	172	292	294	390	446
LPDS Head	186	260	286	481	515	730
Midget Imp.	17	9.5	18	22	31	28
LPDS	112	226	354	405	446	440
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	6.35	6.47	8.55	8.4	9.85	10.6
	Dust Concentration by Mass; mg/m^3					
Hexhlett	1.39	1.87	2.78	2.65	4.25	5.25
NCB Grav.	1.0	2.0		1.5	5.0	7.0
Electrostatic	2.02	2.09	3.74	3.27	6.90	7.70
High Volume	2.35	2.52	4.27	3.91	6.25	7.61
Medium Volume	2.31	2.03	4.47	4.09	6.39	8.8

TABLE C-19
Concentration Measurements
Material: Glass Fibre: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm^3/hour 2.88		6.6		16.1	
	Non Fibrous Dust Concentration by No. p/cm^3 (> 5 µm)					
Midget Imp.	.53	.4	1.08	1.0	5.35	3.55
Midget Imp.	.75	.73	1.18	1.34	5.50	2.55
Konimeters [1]						
Gathercole					1	8.0
Haslam	.52	1	0.8	1.2	1.88	3.56
Sartorius	.80	1.56	2.08	.92	1.4	1.35
Sartorius	1.12	1.78	2.4	1.6	3	1.0
STP [2]	.5	.95	.69	9	.2	4.40
LPDS [3] Head		3.9			1.5	1.4
Midget Imp.	.05	.05		.1	6	1.33
LPDS	0	.62			2	5.6
	Dust Concentration by Number; p/cm^3 (< 5 µm)					
Midget Imp.	7.95	8.85	15.2	12.9	74.9	40.5
Midget Imp.	7.50	8.6	16.3	14.4	79.4	42.6
Konimeters						
Gathercole	456	224	518	188	742	464
Haslam	174	274	515	381	1531	285
Sartorius	222	133	487	245	803	540
Sartorius	226	372	255	888	1124	780
Midget Imp.	5.30	5.43	10.1	8.63	45.0	27.0
	Dust Concentration by Number; p/cm^3 (1-5 µm)					
STP	9.04	10.5	4.5	13.5	.8	18.48
LPDS Head		10			25	5.6
LPDS	19.5	4.5	10.1		9.8	36
	Dust Concentration by Number; p/cm^3 (1/2-5 µm)					
STP			28.5		13	97.7
LPDS Head					134	55
LPDS					325	
	Fibrous Dust Concentration by Number; p/cm^3					
Midget Imp.	6.44	8.0	19.7	13.6	46	39.8
Midget Imp.	8.32	7.36	19.2	15.9	39.2	43.7
Konimeters						
Gathercole	15.0		13	1.3	1	7.6
Haslam	2.68	1.08	1.9	2.2	6.64	8.8
Sartorius	.52		1.64	1.08	3	9.05
Sartorius	1.84		1.7	13	1	5.2
STP	16	16.5	39	24	13	171.6
LPDS Head	24.84	30	78.5	49	139	148
Midget Imp.	5.19	5.43	10.1	10.6	30.8	26.4
LPDS	26.26	22			237	101
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	5.30	4.80	7.40	4.25	6.70	6.95
	Dust Concentration by Mass; mg/m^3					
Hexhlett	.27	.31	.39	.42	.81	1.10
NCB Grav.	.25	.25	.8	1.0	3.0	2.0
Electrostatic	.91	.786	1.59	1.8	2.02	5.72
High Volume	.924	1.03	1.71	1.5	2.64	5.05
Medium Volume	.710	.880	1.36	1.41	2.03	4.10

TABLE C-20
Concentration Measurements
Material: Glass Fibre: Dispersion Method: Pulverized

Sampler	Dust Feed Rate cm^3/hour					
	6.6	16.1	16.1	57		
	Non Fibrous Dust Concentration by No.p/cm^3 (> 5 μm)					
Midget Imp.			4.5	6.5		
Midget Imp.			2.2	12		
Konimeters[1]						
Gathercole						
Haslam						
Sartorius	4.68		5.76	2.24		
Sartorius	9.88		5.76	4.88		
STP[2]	1.6			4.1		
LPDS[3] Head	1					
Midget Imp.			.6	.5		
LPDS	1		5	6.5		
	Dust Concentration by Number;p/cm^3 (< 5 μm)					
Midget Imp.			28	.25		
Midget Imp.			30	49		
Konimeters						
Gathercole						
Haslam						
Sartorius	605		787	925		
Sartorius	1423		1528	714		
Midget Imp.			20	32		
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP	16		10	50		
LPDS Head	27		43	37		
LPDS	20		20			
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	92		79	153		
LPDS Head	141		233	147		
LPDS	70		234	147		
	Fibrous Dust Concentration by Number; p/cm^3					
Midget Imp.			19	11		
Midget Imp.			15	19		
Konimeters						
Gathercole						
Haslam						
Sartorius	1.36		6.32	12.0		
Sartorius	1.16		17.64	13.36		
STP	48		92	105		
LPDS Head	59		183	200		
Midget Imp.	29		14	15		
LPDS			150	183		
	Dust Concentration by Light Scatter; degrees					
Tyndalloscope	5.65	7.9	10.25	10.15		
	Dust Concentration by Mass; mg/m^3					
Hexhlett	.72	.6	1.24	1.9		
NCB Grav.	1.45	.8	1.0	3.2		
Electrostatic	.82	1.46	2	3.3		
High Volume	1.04	1.25	2	4.2		
Medium Volume	1.11	1.3	2	4.0		

TABLE C-21
Dust Concentration in Aggregated Dust Cloud
Material: Coal: Dispersion Method: Atomizer: Full Strength Suspension

Sampler	Volume of Suspension ml 10	6	3
	Dust Concentration by Number Total Count p/cm³ (> 5 µm)		
Midget Imp.	49	18	18.1
Midget Imp.	44	38.8	10.7
Konimeters [1]			
Gathercole		6	8
Haslam	6.5	6.5	6
Sartorius		10	9
Sartorius	7.5	10	6
STP [2]	96	50.4	19.3
LPDS [3] Head	156	55	17.5
Midget Imp.	7.8	5.2	2.6
LPDS	54.1	12.4	20.7
			(< 5 µm)
Midget Imp.	1640	1060	538
Midget Imp.	1680	1120	510
Konimeters			
Gathercole	THTC [4]	1860	1050
Haslam	1550	1200	520
Sartorius	THTC	1850	800
Sartorius	3000	2050	950
Midget Imp.	1388	792	318
			(1-5 µm)
STP	892	492	207
LPDS Head	1166	570	203
LPDS	989	495	261
			(1/2-5 µm)
STP	1324	808	348
LPDS Head	2078	953	386
LPDS	1646	789	426

Footnotes on Page C-2

TABLE C-21
Continued

Sampler	Volume of Suspension ml		
	10	6	3
	Dust Concentration by Number: Aggregates[7] 2 or 3 Particles a/cm^3 (> 5 μm)		
STP	28	16.7	3.8
LPDS Head	78	17.4	10.2
LPDS	21	4.1	0
			(1-5 μm)
STP	238	109	38
LPDS Head	250	122	26.2
LPDS	195	49.5	37.2
			(1/2-1 μm)
STP	8.6	0	0
LPDS Head	0	0	0
LPDS	0	0	0
	Dust Concentration by Number: Aggregates, 4+ Particles a/cm^3 (> 5 μm)		
STP	39	8.7	4.6
LPDS Head	0	0	0
LPDS	4.1	0	0
			(1-5 μm)
STP	8.6	1.1	0.4
LPDS Head	0	0	0
LPDS Head	0	0	0
			(1/2-1 μm)
STP	0	0	0
LPDS Head	0	0	0
LPDS	0	0	0
	Dust Con. by Light Scatter: Degrees		
Tyndalloscope	7.9	4.9	TLTM[6]
	Dust Concentration by Mass, mg/m^3		
Hexhlett	9.24	5.06	2.37
NCB Grav.	7.34	4.27	1.87
Medium Volume	15.6	8.74	4.0

Footnotes on Page C-2

TABLE C-22
Dust Concentration in Aggregated Dust Clouds
Material: Coal: Dispersion Method: Atomizer - Dilute Suspension

Sampler	Volume of Dilute Suspension ml 80	40	20	10
	Dust Concentration by Number: Total Count p/cm³(> 5 μm) [6]			
Midget Imp.	49.2	49.2	13.0	13.8
Midget Imp.	41.4	39	10.7	10.8
Konimeters [1]				
Gathercole				
Haslam		2	7.5	8.2
Sartorius		11		5.0
Sartorius	10		6.2	4.8
STP [2]		39.2	26	16.7
LPDS [3] Head	107	62.5	21.1	18.6
Midget Imp.	7.8	2.6	0	1.7
LPDS	25.5	4.1	12.4	16.5
	(< 5 μm)			
Midget Imp.	2038	1420	717	363
Midget Imp.	1990	1420	775	344
Konimeters				
Gathercole				
Haslam	THTC [4]	2000	1150	620
Sartorius	THTC	THTC	2050	1100
Sartorius	5500	3400	2150	1350
Midget Imp.	1910	995	621	286
	(1-5 μm)			
STP	THTC	1100	596	283
LPDS Head	2390	1060	431	215
LPDS	2140	1040	665	328
	(1/2-5 μm)			
STP	THTC	2210	1181	639
LPDS Head	4715	2230	856	461
LPDS	4110	2300	1345	704

TABLE C-22
Continued

Sampler	Volume of Suspension ml 80	40	20	10
	Dust Concentration by Number Aggregates[7], 2 or 3 Particles a/cm^3 (> 5µm)			
STP		11.2	9.6	5.2
LPDS Head	3.9	3.9	6.6	4.3
LPDS	4.6	0	0	4.1
				(1-5 µm)
STP		287	123	25.0
LPDS Head	364	106	44.8	17.2
LPDS	288	124	62	8.2
				(1/2-1 µm)
STP		0	0	0
LPDS Head	0	0	0	0
LPDS	0	0	0	0
	Dust Concentration by Number Aggregates[7], 4+ Particles, a/cm^3 (> 5 µm)			
STP		10.1	1.4	0
LPDS Head	4.8	7.8	0	0
LPDS	4.6	0	0	0
				(1-5 µm)
STP	THTC[4]	2.2	0	0
LPDS Head	0	0	0	0
LPDS	0	0	0	0
				(1/2-1 µm)
STP	THTC	0	0	0
LPDS Head	0	0	0	0
LPDS	0	0	0	0
	Dust Con. by Light Scatter: degrees			
Tyndalloscope	10.6	9.6	7.2	6.0
	Dust Concentration by Mass, mg/m^3			
Hexhlett	13.95	8.11	4.32	2.12
NCB Grav.	12.0	6.4	4.26	3.0
Medium Volume	18.3	11.38	6.15	3.28

Footnotes on Page C-2

TABLE C-23
Concentration Measurements: Extension to Low Concentrations
Material: Coal: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm^3/hour					
	0.125	0.36	0.51	0.72	1.025	2.88
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	46	50	161	73	237	388
Midget Imp.	46	62	179	75.5	274	410
Konimeters [1]						
Gathercole	504	1065	1461	1600	2160	3204
Haslam	17	44	567	74	966	1591
Sartorius	410	888	1632	1200	2851	3602
Sartorius	522	900	1733	1296	3109	5133
Midget Imp.	37	52	167	69	174	351
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP [2]	68	106	236	106	368	610
LPDS Head [3]	57	160				580
LPDS	73	112		160	372	710
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	266	474	967	522	1508	2630
LPDS Head	270	800				1995
LPDS	312	418		680	1288	2945
	Dust Concentration by Mass; mg/m^3					
Hexhlett	0.17	0.36	1.0	0.444	1.70	2.30
Medium Volume	0.23	0.49	1.23	0.55	1.99	2.92

Footnotes on Page C-2

TABLE C-24
Concentration Measurements: Extension to Low Concentrations
Material: Coal: Dispersion Method: Ejector - No Cyclone

Sampler	Dust Feed Rate cm^3/hour					
	0.26	0.51	1.025	2.88	6.6	
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.	21	45	63		272	
Midget Imp.	28	49	74		272	
Konimeters[1]						
Gathercole	140	256	335	1001	1324	
Haslam	6	9		50	75	
Sartorius	250	358	600	1382	2200	
Sartorius	322	472	624	2179	2461	
Midget Imp.	19	40	59		204	
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP[2]	16.4	28	70	213	288	
LPDS Head[3]		28	76	213	161	
LPDS		33	91	161	201	
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP	36	64	141	366	567	
LPDS Head		57	168	378	452	
LPDS		78	142	270	505	
	Dust Concentration by Mass: mg/m^3					
Hexhlett	0.152	0.294	0.706	1.65	2.73	
Medium Volume	0.29	0.506	1.14	3.77	5.41	

Footnotes on Page C-2

TABLE C-25
Concentration Measurements: Extension to Low Concentrations
Material: Silica: Dispersion Method: Jet Mill

Sampler	Dust Feed Rate cm^3/hour					
	0.1	0.13	0.17	0.26	0.36	0.72
	Dust Concentration by Number; p/cm^3 (< 5 μm)					
Midget Imp.						176
Midget Imp.						187
Konimeters[1]						
Gathercole	316	221	1005	1345	2180	2531
Haslam	124	2		611	1594	2218
Sartorius	325	781		1706	3041	3478
Sartorius	395	900		2223	4143	4578
Midget Imp.						168
	Dust Concentration by Number; p/cm^3 (1-5 μm)					
STP[2]						505
LPDS Head[3]						635
LPDS						900
	Dust Concentration by Number; p/cm^3 (1/2-5 μm)					
STP						1905
LPDS Head						2210
LPDS						2845
	Dust Concentration by Mass; mg/m^3					
Hexhlett	.092	.149	0.8	1.17	2.51	3.17
Medium Volume	.133	.242	1.01	1.57	3.46	4.23

Footnotes on Page C-2

TABLE C-26
Comparison of Dust Sampling Instruments and
Microscope Counting Techniques

Dust Type and Method of Dispersion	Run No.	Dust Concentration by Number (p/cm³) >5 µm					
		Instruments					
		LPDS (Head)				Imp.	Kon.
		Microscopic Techniques					
		tp < $\frac{1}{2}$	tp<1	imp.	kon.	imp.	kon.
Coal							
Jet Milled	165	1430	465	977	1127	241	2181
Jet Milled	168	519	187	440	358	137	1218
Pulverised	62	1184	384	616	1055	134	1000
Pulverised	124	943	230	790	821	118	1372
Ejector Cyclone	58	1112	556	694		178	890
Ejector Cyclone	118	197	116	131	197	86	769
Ejector No Cyclone	121	584	355	507	611	224	1002
Ejector No Cyclone	66	965	470	725	476	165	761
Silica							
Jet Milled	153	1375	378	747	920	198	1281
Jet Milled	150	2448	829	1785	1880	431	2495
Pulverised	112	768	224	490	680	161	998
Pulverised	113	690	204	378	545	188	908
Ejector Cyclone	26	585	337	425	515	210	737
Ejector Cyclone	105	777	332	650	855	314	1371
Ejector No Cyclone	28	1330	760	1161		575	1610
Ejector No Cyclone	109	221	98	129	205	89	464
Pyrite							
Jet Milled	161	2047	156	2309	710	423	2454
Jet Milled	162	1681	153	2096	574	389	2687
Pulverised	97	995	145	639	637	137	774
Pulverised	98	1258	138	735	858	142	637
Ejector Cyclone	41	1398	915	1083	1355	170	1468
Ejector Cyclone	102	148	59	115	127	107	387
Ejector No Cyclone	101	201	81	184	212	127	475
Ejector No Cyclone	100	728	315	781	673	369	1220
Mica							
Jet Milled	170	1139	355	633	844	473	2004
Jet Milled	172	2175	735	1572	1450	718	2668
Pulverised	176	1214	269	930	1632	344	1742
Pulverised	71	1948	480	1295	1320	235	1588
Ejector Cyclone	183	568	275	372	678	321	1464
Ejector Cyclone	78	2070	430	595	1158	508	1875
Ejector No Cyclone	79	1876	426	870		526	1697
Ejector No Cyclone	178	1096	431	736	752	512	2169

TABLE C-26
Continued

		Dust Concentration by Number $(p/cm^3) > 5\mu m$					
		Instruments					
		LPDS Head				Imp.	Kon.
		Microscopic Techniques					
		Particles and Fibres					
Asbestos							
Jet Milled	141	713		162	273	113	1391
Jet Milled	143	945		271	423	246	1915
Pulverised	85	1345		427	576	256	2717
Pulverised	86	1838		694	443	526	2646
Glass Fibre							
Jet Milled	144	273		234	183	105	1050
Jet Milled	148			44	61	14	269
Pulverised	90	347		268	295	52	820
Pulverised	91	373		249	224	42	1157
		Fibres Only					
Asbestos							
Jet Milled	141	620		65	51	23.9	2.74
Jet Milled	143	770		167	278	47.4	1.49
Pulverised	85	226		68	49	11.2	1.30
Pulverised	86	405		209	95	23.3	2.95
Glass Fibre							
Jet Milled	144	139		76	13	38.7	2.9
Jet Milled	148	25		18	19	6.6	5.0
Pulverised	90	200		62	31	15.0	12.0
Pulverised	91	183		64	26	16.0	11.98

APPENDIX D
COMPARISON OF SIZE SELECTORS
TABLES OF RESULTS

These tables list the detailed results obtained in comparisons between dust samplers fitted with respirable dust size selectors. Table D-1 gives the results of the first set of comparisons between horizontal elutriators and 10 mm nylon cyclone size selectors.

Tables D2 to D-11 give the results obtained in the main experiment in which various cyclones and size selectors and construction details of the horizontal elutriator samplers were examined.

FOOTNOTES TO TABLES

1. Hexhlett operated as supplied by Casella with critical-flow control orifice between elutriator plates and filter.

2. Hexhlett operated for shorter time than other instruments - results not comparable with them.

3. Hexhlett modified by removal of critical orifice from between elutriator plates and filter. Another flow control placed after filter.

4. Laboratory sampler 1 built to similar specifications as Casella type 113A but without turned-up lip on ends of elutriator plates.

5. As LS 1 but with a 0.2-inch-diameter orifice between elutriator plates and filter.

6. As LS2 but with a 0.1-inch-diameter orifice.

7. Casella personal sampler as supplied.

8. Casella personal sampler modified by removing one rubber finger cot and restrictive orifice from flow smoother.

9. Flow obtained from Casella personal sampler pump with smoother.

10. Laboratory sampler.

11. Laboratory sampler upside down.

12. Unico 0.5 inch cyclones, stainless steel sheet.

TABLE D-1
Dust Concentration Measurements Using Elutriator and Cyclone Size Selectors

Dust Cloud	Respirable Dust Concentration mg/m³							
	Horizontal Elutriators				Cyclones			
	5 µm	MRC Standard						
		Airflow ℓ/min						
	25	50[1]	2.83	2.5	2.64	1.95	1.65	1.3
Coal								
Jet Milled	15.5	16.8	17.8	17.9	10.9	17.1		18.0
	19.5	21.4	20.6	22.6	16.6	25.0		25.6
Cyclone	14.1	20.3	18.5	20.2	10.1	13.6		22.9
	15.3	21.1	21.2	24.0	13.2	16.4		24.9
Silica								
Jet Milled	14.35	16.1	18.8	17.5	12.2	15.4	15.3	18.1
	10.6	11.9	12.1	12.2	9.0	11.2	10.2	13.7
	9.90	10.75	13.0	11.7	10.3	13.4	13.5	14.2
	10.9	12.1	13.4	13.4	8.3	11.5	13.6	15.0
Pulverised	13.0	19.0	17.1	18.4	10.7	14.6	16.7	19.6
	12.9	18.5	17.7	18.2	9.95	13.8	15.8	20.2
Cyclone	11.1	18.9	16.3	17.0	7.8	12.3	13.6	16.2
	10.6	15.5	15.6	14.6	7.8	10.5	13.2	15.8
	5.3	6.7	7.0		4.9	6.4	6.8	7.1
	8.8	11.8	11.5	12.6	7.1	8.6	10.2	11.9
No Cyclone	10.9	16.3	16.0	17.1	9.1	11.9	13.6	18.8
	10.9	16.4	15.8	16.1	7.9	11.8	12.1	16.9
Pyrite								
Jet Milled	43.8	52.7	46.5	53.4		45.7	47.4	53.1
	18.7	21.0	21.7	21.6	16.0	18.8	20.0	24.4
Cyclone	3.3	5.3	5.5	5.6	2.75	3.9	4.4	6.35
	3.8	6.0	5.6	5.25	3.1	4.4	4.6	5.6
	3.5	5.7	5.16	5.8	2.4	3.3	3.6	4.7
Mica								
Jet Milled	16.1	19.8	19.0	20.3	15.2	16.2	19.6	20.3
	17.05	20.95	21.95	23.3			22.2	23.9
Glass Fibre								
Jet Milled	3.4	3.9	3.7	3.7	2.8	3.0	3.4	4.0
Pulverised	1.6	2.25	1.85	1.9	1.45	1.7	2.3	2.3
	.97	1.21	.91	1.1	.48	.8	.77	1.1
Asbestos								
Jet Milled	12.6	13.6	13.75	12.0	11.8	12.05	11.8	11.2
	12.5	13.3	12.7	13.0	9.5	11.5	12.2	12.7
Pulverised	4.2	5.4	4.9	5.4	2.8	4.2	4.2	4.8
	5.2	6.6	6.0	6.6	4.3	5.0	5.0	6.2

Footnotes on Page D-2

TABLE D-2
Dust Concentration Measurements
Material: Coal: Dispersion Method: Jet Mill

Size Selectors	Air Flow ℓ/min	Run Number 7	8	9	10	11	12	13	14
		Respirable Dust Concentration mg/m³							
Elutriators									
Hexhlett [2]									
Standard	50	38.1	39.4	36.3	42.7	54.8	55.8	46.7	56.4
Modified [3]	50	39.5	42.2	38.4	46.3	58.7	58.7	50.5	59.4
Casella									
Type 112A	2.5		38.0		43.6			47.0	
Upside down	2.5	43.6		41.2			51.2		52.4
Lab. Built									
I [4]	2.83	45.2	40.6	39.3	46.6	50.0	55.4	47.5	54.8
II [5]	2.83		36.0		40.9	41.6		44.0	
III [6]	2.83	37.5		40.9			49.5		48.3
Cyclones									
10 mm Nylon									
A	1.28	30.9				41.0			
	1.68				30.4				43.4
	1.92			22.4				35.2	
	1.77 [9]		28.9				41.3		
B	1.28		35.9				38.9		
	1.68	32.4				36.4			
	1.92				33.0				40.4
	1.77 [9]			28.6				37.6	
C	1.28			35.5				45.6	
	1.68		32.4				46.7		
	1.92	34.4				41.2			
	1.77 [9]				37.0				45.8
D	1.28				41.9				54.0
	1.68			31.8				39.6	
	1.92		30.0				42.2		
	1.77 [9]	32.8				34.1			
Casella									
Complete [7]	1.83 [9]		40.0		46.2	53.3	61.5		
Modified [8]	1.90 [9]	42.7						49.2	61.3
		Total Dust Concentration mg/m³							
Open Filters									
	2.63	46.4	45.0	45.0	51.7	54.5	68.2	53.6	67.2
	3.41	46.6	45.5	44.0	51.5	55.5	65.8	53.9	66.3

Footnotes on Page D-2

TABLE D-3
Dust Concentration Measurements
Material: Coal: Dispersion Method: Ejector - No Cyclone

Size Selectors	Air Flow ℓ/min	Run Number 15	16	17	18	19
		Respirable Dust Concentration mg/m^3				
Elutriators						
Hexhlett [2]						
Standard	50	47.9		36.9	30.3	34.1
Modified [3]	50	49.6		41.6	34.3	32.3
Casella						
Type 112A	2.55				40.0	
Upside down	2.55		39.4	40.4		30.8
Lab. Built						
I [4]	2.83	46.0	43.6	45.7	37.8	34.4
II [5]	2.83		32.7	42.8		32.5
III [6]	2.83	40.4			32.4	
Cyclones						
10 mm Nylon						
A	1.28	36.8		41.5		
	1.68					26.0
	1.92				30.6	
	1.77 [9]		34.7			
B	1.28		40.6			
	1.68	30.8		31.0		
	1.92					22.3
	1.77 [9]				27.5	
C	1.28				33.1	
	1.68		34.8			
	1.92	29.7		28.4		
	1.77 [9]					27.1
D	1.28					31.1
	1.68				26.5	
	1.92		27.3			
	1.77 [9]					
Casella						
Complete [7]	1.83 [9]			56.3		44.8
Modified [8]	1.90 [9]	51.0	51.0		48.3	
		Total Dust Concentration mg/m^3				
Open Filters	2.63	92.5	75.4	111.0	116.9	90.2
	3.41	96.8	91.2	111.0	98.8	90.5

Footnotes on Page D-2

TABLE D-3
continued

Size Selectors	Air Flow ℓ/min	Run Number			
		20	21	22	23
		Respirable Dust Concentration mg/m³			
Elutriators					
Hexhlett [2]					
Standard	50	29.8	39.2	36.9	33.4
Modified [3]	50	33.6	39.8	39.2	41.4
Casella					
Type 112A	2.55		45.4		47.2
Upside down	2.55	44.0		50.0	
Lab. Built					
I [4]	2.83	38.4	40.8	43.1	43.9
II [5]	2.83	33.0		38.6	
III [6]	2.83		36.4		
Cyclones					
10 mm Nylon					
A	1.28	35.5			
	1.68				35.2
	1.92			32.7	
	1.77 [9]		32.4		
B	1.28		34.2		
	1.68	27.2			
	1.92				29.2
	1.77 [9]			37.4	
C	1.28			37.4	
	1.68		30.9		
	1.92	28.1			
	1.77 [9]				35.0
D	1.28				37.5
	1.68			41.2	
	1.92		32.7		
Casella					
Complete [7]	1.83 [9]	45.6		54.6	
Modified [8]	1.90 [9]		51.9		54.2
		Total Dust Concentration mg/m³			
Open Filters					
	2.63	70.7	83.1	80.3	
	3.41	73.9	83.2	84.2	92.5

Footnotes on Page D-2

TABLE D-4
Dust Concentration Measurements
Material: Coal

Size Selector	Air Flow ℓ/min	Dispersion Method: Ejector - No Cyclone				Dispersion Method: Jet Mill			
		Run Number							
		24	25	26	27	28	29	30	31
		Respirable Dust Concentration mg/m³							
Elutriators									
Hexhlett [2]									
Standard	50	6.58	9.62	7.57	6.59	15.8	15.1	17.0	18.7
Modified [3]	50	6.83	10.33	7.29	7.05	16.4	17.9	18.1	19.2
Casella									
Type 112A	2.55		10.61	7.34	6.96	15.9	18.0	17.9	18.8
Lab. Built									
I [4]	2.83	7.1	9.95	7.15	7.1	16.1	18.2	17.8	19.3
II [5]	2.83		9.0		6.52		15.8		17.1
III [6]	2.83	5.61		6.22		14.1		15.4	
		Total Dust Concentration mg/m³							
Open Filters	2.63	19.5	28.1	20.2	19.5	22.7	25.8	25.8	28.4

Footnotes on Page D-2

TABLE D-5
Dust Concentration Measurements
Material: Coal: Dispersion Method: Jet Mill

Size Selectors	Air Flow ℓ/min	Run Number 43	44	45	46	47	48	58
		Respirable Dust Concentration mg/m³						
Elutriators								
Hexhlett Modified [3]	50	69.0	62.0	64.5	59.2	54.9	52.5	29.4
Casella Type 112A	22.5		56.0	62.4	54.4		46.0	31.7
Lab. Built								
I [4]	2.83		60.5	65.8	58.4	55.5	52.5	29.2
II [5]	2.83	69.7		67.0		54.3		
III [6]	2.83		57.8		54.3		50.5	29.7
Cyclones								
10 mm Nylon								
A	1.28							27.9
B	1.28			67.6			46.0	
	1.68	59.5			61.0			23.8
	1.92		49.0			42.2		
C	1.28		62.5			51.5		
	1.68			56.7			48.8	
	1.92	57.5			54.0			26.1
D	1.28	60.4			55.0			
	1.68		50.3			45.8		
	1.92			57.4			43.0	25.5
Casella								
Complete [7]	1.83 [9]	80.5		74.3		61.0		
Modified [8]	1.90 [9]		64.5		66.3		58.7	
		Total Dust Concentration mg/m³						
Open Filters								
	3.41	82.6	72.6	79.8	71.4	66.2	63.6	

Footnotes on Page D-2

TABLE D-6
Dust Concentration Measurements
Material: Coal: Dispersion Method: Ejector - No Cyclone

Size Selector	Air Flow ℓ/min	49	50	51	52	53
		Respirable Dust Concentration mg/m^3				
Elutriators						
Hexhlett						
Modified [3]	50	46.3	47.1	45.8	46.8	43.3
Casella						
Type 112A	2.5	51.2	48.1	45.8	49.1	46.7
Lab. Built						
I	2.83	45.6				
IV	2.83	47.7		43.5		41.3
V upside down	2.83		44.5		44.4	
Cyclones						
10 mm Nylon						
A	1.28	48.1				38.3
	1.68				36.9	
	11.92			33.6		
	1.95		28.6			
B	1.28		39.9			
	1.68	33.1				29.4
	1.92				32.6	
	1.95			27.7		
C	1.28			41.5		
	1.68		35.5			
	1.92	32.8				30.5
	1.95				37.2	
D	1.28				45.8	
	1.68			35.4		
	1.92		31.3			
	1.95	30.3				27.2
Casella						
Complete [7]	1.85 [9]	59.4		55.1		54.7
Modified [8]	2.07 [9]		51.8		52.8	
		Total Dust Concentration mg/m^3				
Open Filters	3.41	95.7	95.8	92.0	103.5	90.6

Footnotes on Page D-2

TABLE D-6
continued

Size Selector	Air Flow l/min	Run Number 54	56	57	60	61
		Respirable Dust Concentration mg/m³				
Elutriators						
Hexhlett						
Modified [3]	50	57.2	57.7	49.5	58.6	47.9
Casella						
Type 112A	2.5	60.8	54.0	49.5	61.5	48.8
Lab. Built						
I	2.83	54.2		46.9	56.2	48.2
IV	2.83	55.0		48.7		
V upside down	2.83		50.4		54.5	42.8
Cyclones						
10 mm Nylon						
A	1.28				58.6	42.2
	1.68			36.1		
	1.92		36.5			
	1.95	40.6				
B	1.28	50.0				
	1.68				41.2	32.0
	1.92			33.1		
	1.95		33.4			
C	1.28		46.5			
	1.68	42.3				
	1.92				36.6	32.3
	1.95			37.1		
D	1.28			49.0		
	1.68		39.8			
	1.92	42.5				
	1.95				35.2	31.8
Casella						
Complete [7]	1.85[9]		63.4			
Modified [8]	2.09[9]	62.4		53.9	65.6	55.4
		Total Dust Concentration mg/m³				
Open Filters	3.41	119.8	105.3	103.0	117.2	110.0

Footnotes on Page D-2

TABLE D-7
Dust Concentration Measurements
Material: Coal: Dispersion Method: Ejector - No Cyclone

Size Selectors	Air Flow ℓ/min	Run Number							
		65	66	67	68	69	70	71	72
		Respirable Dust Concentration mg/m³							
Elutriators									
Hexhlett									
Modified [3]	50	46.2	45.2	46.4	49.1	48.7	49.3	49.3	49.0
Casella									
Type 112A	25	47.5	47.8	49.5	49.8	48.4	52.4	51.8	51.8
Lab. Built									
I	2.83	47.6	48.4	50.4	49.7	47.5	49.7	49.3	49.4
IV	2.83		42.2		46.6		49.3		48.6
V	2.83	44.3		46.5		46.0		46.4	
Cyclones									
10-mm Nylon									
C	1.28	40.6				43.4			
	1.68				34.2				33.9
	1.92			34.0				29.6	
	1.95		31.0				33.1		
D	1.28		39.4				46.1		
	1.68	34.8				37.2			
	1.92				36.2				32.6
	1.95			32.1				32.1	
E	1.28			39.1				42.1	
	1.68		27.4				34.4		
	1.92	28.4				31.7			
	1.95				38.8				29.6
F	1.28				46.5				43.0
	1.68			38.7				36.7	
	1.62		22.4				36.5		
	1.95	30.5				32.6			
Casella									
Complete [7]	1.85	57.5		60.3			61.2		62.2
Modified [8]	2.1		50.7		54.1	57.9		58.1	
0.5-in. S.S.									
A	8.21	50.0		62.5			68.0		63.8
	9.95		59.7		56.7	58.9		55.2	
B	8.21		56.7		62.8	64.2		61.6	
	9.95	52.1		62.0			61.7		60.6
		Total Dust Concentration mg/m³							
Open Filters	3.41	97.2	96.5	99.0	103.7	102.0	107.1	103.4	102.1

Footnotes on Page D-2

TABLE D-8
Dust Concentration Measurements
Material: Coal: Dispersion Method: Jet Mill

Size Selectors	Air Flow ℓ/min	Run Number							
		73	74	75	76	77	78	79	80
		Respirable Dust Concentration mg/m³							
Elutriators									
Hexhlett									
Modified [3]	50	24.5	29.2	36.7	38.25	39.5	37.8	36.6	38.7
Casella									
Type 112A	2.55	26.6	30.1	38.9		40.4			
Lab. Built									
I	2.83	23.9	29.5	36.5	37.4	40.4	37.4	35.8	38.7
IV	2.83	24.9		34.6		39.5		37.0	
V	2.83		26.9		36.5		35.0		38.0
Cyclones									
10-mm Nylon									
C	1.28	21.5				36.8			
	1.68				31.5				32.1
	1.92			29.5				27.7	
	1.95		23.4				30.8		
D	1.28		27.0				37.8		
	1.68	1.99				33.4			
	1.92				31.3				31.6
	1.95			28.1				27.4	
E	1.28			35.4				35.7	
	1.68		23.0				33.1		
	1.92	21.6				30.5			
	1.95				28.8				29.5
F	1.28				35.7				35.2
	1.68			31.8				28.9	
	1.92		22.7				30.1		
	1.95	20.5				29.0			
Casella									
Complete [7]	1.85	28.1		40.8			42.8		45.3
Modified [8]	2.1		30.9		39.4			42.1	
0.5-in. S.S.									
A	8.21	23.6					36.4		33.8
	9.95		25.5		33.8	35.1		31.6	
B	8.21		27.4		36.7	35.8		35.0	
	9.95	22.1					33.0		36.7
		Total Dust Concentration mg/m³							
Open Filters									
	3.41	29.2	34.9		46.2	47.8	45.8		46.1

TABLE D-9
Dust Measurements at Low Concentrations
Material: Coal

Size Selectors	Air Flow l/min	Dispersion Method Jet Mill Run Number 81	83	Ejector - No Cyclone 84	85	86	87
		Respirable Dust Concentration mg/m^3					
Elutriators							
Hexhlett							
Modified [3]	50	11.3	5.65	40.2	6.81	11.4	11.7
Casella							
Type 112A	2.55	12.1	6.01	43.4	7.25		
Lab. Built .							
I	2.83	10.8	5.52	41.5	7.03	11.3	12.0
IV	2.83	10.8		42.1	6.97	11.1	11.9
Cyclones							
10-mm Nylon							
C	1.28	10.5		36.2	5.73	8.83	10.1
	1.93		4.12				
D	1.28		5.60				
	1.68	9.01		26.0	4.48	7.08	8.21
E	1.68		4.20				
	1.75	8.7		21.2	4.16	6.74	7.16
F	1.75		4.35				
	1.93	7.81		23.5	3.83	6.78	7.72
Casella							
Complete [7]	1.85	15.1	7.2	50.6	8.76		15.3
0.5-in. S.S.							
A	8.21		5.4		7.98		
	9.95	11.4				13.7	13.9
B	8.21	11.4					
	9.95		4.94		7.93		
		Total Dust Concentration mg/m^3					
Open Filters	3.41	16.8	7.71	81.5	13.9	23.7	25.1

Footnotes on Page D-2

TABLE D-10
Dust Concentration Measurements
Material: Coal: Dispersion Method: Jet Mill

Size Selector	Air Flow ℓ/min	Run Number 105	106	107	108	109	110	111	112
		Respirable Dust Concentration mg/m³							
Elutriators									
Hexhlett Modified [3]	50	19.6	21.0	25.4	21.1	18.8	20.3	18.1	20.9
Casella Type 112A	2.55	19.3	22.0	25.5	21.9	18.3	22.3		
Cyclones									
10-mm Nylon									
C	0.99	17.2	21.8			17.2			
	1.52				19.7			14.9	
	1.71			22.9			17.9		18.5
D	0.99			24.9			21.1		22.6
	1.52	17.7	19.1			17.6			
	1.71				17.5			15.6	
E	0.99				22.0			16.6	
	1.52			21.1			17.1		18.6
	1.71	15.2	17.3			14.9			
Casella									
O	1.91							19.0	23.0
	2.39			2.44			21.0		
	2.82	16.4	16.8			15.8			
1	1.91	19.1	21.6			19.0			
	2.39				19.4			15.5	18.0
	2.82			18.8			16.4		
2	1.91			28.9			23.4		
	2.39	17.7	20.6			17.7			
	2.82				18.1			13.6	16.9
3	2.1	20.3	22.4	23.0	22.0	20.5	22.2	17.5	20.3
4	2.1	18.4	20.5	25.4	22.6	21.3	22.1	18.3	19.8
0.5-in. S.S.									
A	8.15	18.2	19.7		19.6	17.7	20.0		
	16.15			20.2				14.3	16.2
B	8.15			26.8				16.6	17.0
	16.15		15.45		15.75	13.3	16.3		

Footnotes on Page D-2

TABLE D-11
Dust Concentration Measurements
Material: Coal: Dispersion Method: Ejector - No Cyclone

Size Selectors	Air Flow ℓ/min	Run Number 113	114	115	116	117
		Respirable Dust Concentration mg/m³				
Elutriators						
Hexhlett						
Modified [3]	50	31.0	31.7	24.6	31.8	27.2
Casella						
Type 112A	2.55					
Cyclones						
10-mm Nylon [4]						
C	0.99	33.8			34.0	
	1.52			21.0		
	1.71		22.1			19.1
D	0.99		37.6			30.3
	1.52	34.6			27.4	
	1.71			20.4		
E	0.99			22.7		
	1.52		22.8			19.5
	1.71	17.1			21.2	
Casella						
0	1.91			30.2		
	2.39		28.0			24.5
	2.82	22.9			23.6	
1	1.91	29.6			33.4	
	2.39			18.4		
	2.82		18.0			16.1
2	1.91		35.0			34.4
	2.39	20.2			26.8	
	2.82			17.1		
3	2.1	27.4			29.6	27.4
4	2.1	27.5		23.6	30.5	28.4
0.5-in. S.S.						
A	8.15	33.0		30.3	40.2	
	16.15		26.9			26.0
B	8.15		28.6			37.0
	16.15	27.0			31.4	

Footnotes on Page D-2

TABLE D-11
continued

Size Selectors	Air Flow ℓ/min	Run Number 118	119	120	121
		Respirable Dust Concentration mg/m³			
Elutriators					
Hexhlett Modified [3]	50	30.6	26.0	29.8	26.4
Casella Type 112A	2.55	34.0	29.2	32.7	25.9
Cyclones					
10-mm Nylon					
C	0.99		30.3		
	1.52	25.9			21.5
	1.71			22.8	
D	0.99			34.5	
	1.52		22.4		
	1.71	24.8			20.3
E	0.99	33.4			27.3
	1.52			21.9	
	1.71		18.3		
Casella					
0	1.91	38.4			31.6
	2.39			27.2	
	2.82		18.6		
1	1.91		26.2		
	2.39	24.4			19.7
	2.82			17.6	
2	1.91			32.0	
	2.39		18.8		
	2.82	22.4			17.0
3	2.1	31.4	23.1	27.4	25.0
4	2.1	33.8	24.6	31.0	27.4
0.5-in. S.S.					
A	8.15		31.9		30.4
	16.15	25.9		26.3	
B	8.15	41.7		36.6	
	16.15		27.1		25.3

Footnotes on Page D-2

1. 3

Section 1—Chapter 3

Section 1—Chapter 3

Full-Shift Assessment of Respirable Dust Exposure

by

G. Knight, T.E. Newkirk and G.R. Yourt

Canadian Mining and Metallurgy Bulletin 67(4) 12p (1974)

ÉVALUATION DE L'EXPOSITION À LA POUSSIÈRE INHALABLE PENDANT UN POSTE DE TRAVAIL COMPLET

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RÉSUMÉ

Il n'existe pas de concentration bien définie au-dessous de laquelle on peut considérer que la poussière est inoffensive. Dans les opérations minières, il est impossible d'éviter complètement la production de poussière. Néanmoins, les méthodes utilisées pour lutter contre la poussière ont entraîné une diminution remarquable des maladies. Pour poursuivre dans cette voie et protéger la santé des mineurs, il est nécessaire de mesurer les concentrations de poussière et, dans certains cas, de déterminer les éléments constituants.

Les travaux de recherche et essais sur le terrain exhaustifs menés récemment ont conduit à la mise au point d'instruments et de méthodes qui permettent d'évaluer les dangers associés à l'exposition à la poussière inhalable avec une plus grande précision que ne le permettent les techniques actuelles.

1. L'échantillonneur de poussière inhalable gravimétrique porté par un travailleur ou fixé à proximité, est efficace et permet de recueillir un seul échantillon pour un poste de travail complet. À partir de cet échantillon, on peut déterminer la masse totale ainsi que la quantité de quartz et d'autres particules telles que les particules de fumée diesel.

2. Dans les mines riches en quartz où le poids des échantillons est nécessairement faible, la mesure directe du quartz sur les filtres par diffraction des rayons X est plus précise et plus appropriée que la pesée.

3. Des échantillons conimétriques, dans la fourchette 200-300 $ppcm^3$, recueillis simultanément avec un échantillonneur gravimétrique dans des mines riches en quartz, semblent correspondre à 0,1 mg/m^3 de quartz dans la formule de l'ACGIH relative à la masse - TLV = 10/% qtz.inh. + 2 (en $mg.m^3$).

4. Les filtres des échantillonneurs portés par les travailleurs ont donné des concentrations de poussière inhalable supérieures à celles obtenues avec les échantillonneurs fixés à proximité.

5. Lorsqu'un même travailleur portait deux échantillonneurs gravimétriques, avec un ensemble cyclone-filtre fixé au revers et un autre ensemble fixé à la batterie de la lampe de chapeau, les indications étaient suffisamment proches pour que l'on adopte le dernier mode de fixation.

6. La coopération des mineurs a été excellente; très peu d'échantillons ont été perdus.

7. Les problèmes associés au fonctionnement des échantillonneurs étaient mineurs. Certains ont été éliminés dans les nouveaux appareils fixés à la batterie de la lampe de chapeau.

Full-Shift Assessment of Respirable Dust Exposure

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Abstract

There is no known well-defined level of concentration below which dust may be said to be harmless. In mining operations, the production of dust cannot be entirely avoided. Nevertheless, methods used to control dust have been rewarded by a significant decrease in the incidence of disease. To guide these efforts and safeguard the miners' health it is necessary to measure dust concentrations, and in some cases analyze the constituents.

Extensive recent research and field testing has resulted in the development of instruments and methods that more accurately assess the health hazard from exposure to respirable dust than do current practices.

1. The gravimetric respirable dust sampler worn by a worker, or operated in a fixed position nearby, is effective and convenient for collecting a single sample over an entire shift. From this sample the total mass, the quantity of quartz and other particulates such as diesel smoke can be determined.

2. For high-quartz mines where the weight of samples is necessarily small, direct measurement of quartz on filters by X-ray diffraction is more accurate and suitable than weigking.

3. Konimeter samples in the range of 200 to 300 ppcc, taken simultaneously with a gravimetric sampler in high-quartz mines, appear to correspond with 0.1 mg/m^3 of quartz in the ACGIH mass formula — $TLV = 10/\%$ resp. qtz. + 2 (in mg/m^3).

4. Filters from samplers worn by workmen yielded higher readings of respirable dust than those from samplers in fixed positions nearby.

5. When two gravimetric samplers were worn by the same miner, with one cyclone and filter attached to the lapel and the other to the cap-lamp battery, readings were sufficiently similar to warrant adopting the latter arrangement.

6. The co-operation of miners in wearing samplers was very gratifying; very few samples were spoiled.

7. Problems encountered in the operation of samplers were minor. Some are eliminated in the new units incorporated with the cap-lamp battery.

Geoffrey Knight was born in Oxfordshire, received his early schooling in England and graduated from the University of London in 1946. He was a research assistant at the Kodak Research Laboratories from 1943 to 1953, and then joined the Dust Group of the National Coal Board Mining Research Establishment in Middlesex as a research scientist. Mr. Knight emigrated to Canada in 1965 and joined the Department of Energy, Mines and Resources, Mines Branch, Elliot Lake. He is currently research scientist in charge of the Dust Research Laboratory at the Mining Research Centre, Elliot Lake Laboratory.

Tracy Earle Newkirk was born in St. William, Ontario and attended Queen's University in Kingston.

He joined McIntyre Porcupine Mines Limited in 1929 and left in 1942 to join the army, returning there after the war. He was appointed as ventilation engineer at McIntyre in 1934, then industrial engineering superintendent and later industrial relations superintendent, a position he held until his retirement in November 1973. He is currently technical adviser to the McIntyre Research Foundation.

Mr. Newkirk is a member of the Porcupine Branch of The Canadian Institute of Mining and Metallurgy and the Association of Professional Engineers of the Province of Ontario, and has served on various Mines Accident Prevention Association of Ontario committees.

G. R. Yourt gained his higher learning in the Arts and Sciences at Queen's University (B.A.). He has spent over 35 years in the mining industry with Wright-Hargreaves Mines Ltd. and the Mines Accident Prevention Association of Ontario and, until 1969, was employed by Rio Algom Mines Ltd. as environmental consultant. He is currently working as a private consultant.

Mr. Yourt has devoted major efforts to the health and safety of mining personnel through intensive engineering work in mine ventilation and dust and radiation control. He has had experience in the fields of human relations, supervisory development, administration studies and industrial engineering and as the author of a number of papers covering ventilation and dust control, the prevention of mine fires and the control of radiation.

A Life Member of the Institute, Mr. Yourt is also a member of the Mining Section of the National Advisory Committee on Mining and Metallurgical Research, the Committee on Occupational Health of the MAPAO, the AIHA and the Association of Professional Engineers of Ontario.

Paper Presented: at the 75th Annual General Meeting of the CIM, Vancouver, April 1973.

Keywords: Environmental control, Dust exposure, Industrial hygiene, Gravimetric samplers, Silicosis, Konimeters, Samplers, Respirable dust.

Introduction

THE NEED FOR, the historical development of and the research studies on instrumentation for full-shift sampling of respirable dust at metal mines are described. At the request of and in participation with the Mines Accident Prevention Association of Ontario (MAPAO), the project was undertaken and financed by the McIntyre Research Foundation in co-operation with the Mines Branch, Department of Energy, Mines and Resources (EMR), Ottawa.

Historical Aspects

A brief history of dust sampling and the incidence of silicosis at Ontario mines, the need for full-shift assessment of dust exposure with quartz analysis, and the development of technology and suitable instrumentation for this purpose are discussed in Appendix A.

Research Project, 1970-72

Research on dust sampling described in this paper was initiated early in 1970.

OBJECTIVES

The main objective was to determine the criteria which would enable the Association to recommend to its members the instruments and procedures that should be used at mining operations to assess, as accurately as feasible, the harmfulness as well as quantity of full-shift dust exposures.

A supplementary objective was to develop a more effective and reliable method to measure concentrations of aluminum powder dispersed in change rooms and treatment chambers to help prevent the development of silicosis. As a result of this study a revised procedure was issued by one of the authors(1). It was sent to all users of aluminum powder and is available to interested parties from the Foundation.

ARRANGEMENTS

In 1969 a dust chamber, various sampling instruments and arrangements for testing them had been established and were in use at the Mines Branch's Elliot Lake Research Laboratory. It is located near highly siliceous metal mines where gravimetric dust sampling had been initiated and warranted continuation.

Arrangements were made for a technologist employed by the Foundation to use the Research Laboratory as an operating base under the direct supervision of the research scientist, a co-author. The other two authors participated in the general co-ordination of the studies on behalf of the Foundation and the Association. Helpful suggestions were also contributed by the Association's Sub-Committee on Dust Control and Ventilation, which now, somewhat enlarged, functions as a regular committee.

FIGURE 1A — Personal sampler.

INSTRUMENTS TESTED

A list of dust samplers purchased for testing and other instruments used in the study is provided in Appendix B. All samplers were calibrated in the Laboratory before they were operated in the mines.

LABORATORY TECHNIQUE

Mass measurements were conducted by weighing the filters with a Mettler semimicrobalance, to an accuracy of about 0.05 mg. Quartz analysis was done by X-ray diffraction, as described by Knight(3,4). According to Knight and Leroux(5), the most sensitive method for analyzing quartz is by a direct X-ray reading on dust collected on a silver membrane using the area that fits the beam, about 10 by 20 mm.

The Casella personal samplers, fitted with 25-mm-diameter and 5-μ pore-size silver membrane filters, were used for most of the testing, both for personal and fixed-position sampling. Some of the samples were ashed. Readings were expressed as mg/m^3 (milligrams per cubic metre) of total respirable dust, respirable quartz or respirable combustible particulates (loss in weight by incineration).

The Modified Thermal Precipitator (MTP) dust samples were acid-treated, incinerated, shadowed with metallic aluminum and washed or brushed. The resulting apertures in the aluminized coating were measured by an optical technique known as diffraction size analysis (DISA). The technique is reported by Talbot(6) to provide an estimate of the respirable surface area (RSA) of the ashed dust collected on the slide in square microns per cubic centimeter ($\mu m^2/cm^3$). The DISA readings were processed by the Corner House Laboratories in South Africa. The same samples were also counted at the Elliot Lake Laboratory under a microscope before and after treatment to give estimates of dust concentration by number (p/cm^3 or ppcc) for comparison with the konimeter and Long-Period Dust Sampler.

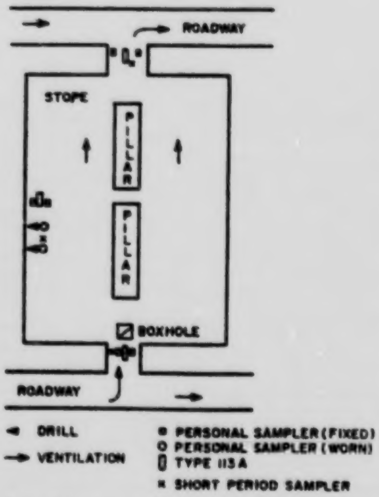


FIGURE 1B — Typical sampling positions.

Konimeter slides were incinerated at the Laboratory, then acid-treated and counted in the Association's North Bay office with the electronic scanner as described by Chmara(7). Usually three spots are taken for each sample and the average is expressed in particles per cubic cm. In this study, the three spots were spread over 5 minutes to provide a comparison with the 5-minute MTP samples.

Further details regarding sampling instruments and techniques used during the research project are available from one of the authors (Knight).

UNDERGROUND SAMPLING

The following standard procedure was usually followed when taking samples underground during the first year of studies.

1. The technologist fitted two miners with personal samplers, as illustrated in Figure 1a, on reaching their working place at the beginning of a shift. Pumps were started at this time and stopped at the end of the shift.
2. In some or all working places tested, samples were taken: (1) in the men's breathing zones (lapel area), (2) at fixed positions as near to the miners as feasible, and (3) in the air entering and (4) in the air leaving the operation. Konimeter and MTP samples were taken at regular intervals throughout the shift by the technologist and his assistant. Figure 1b illustrates the sampling positions at a typical drilling operation. The fixed-position samplers were moved so that they were always within 10 feet of the drillers. At slushing operations the fixed samplers did not include any additional exposure while an operator left the slusher to attend to other duties.

Most of the sampling was done at the actual working sites. Sampling of air entering and of air leaving follows standard practice in semi-annual surveys and is done for control purposes. For example, if concentrations are high for air entering, it partly explains why readings are high at the operations and in air leaving. If concentrations are low in air entering and leaving and high at the operation, distribution of air is poor.

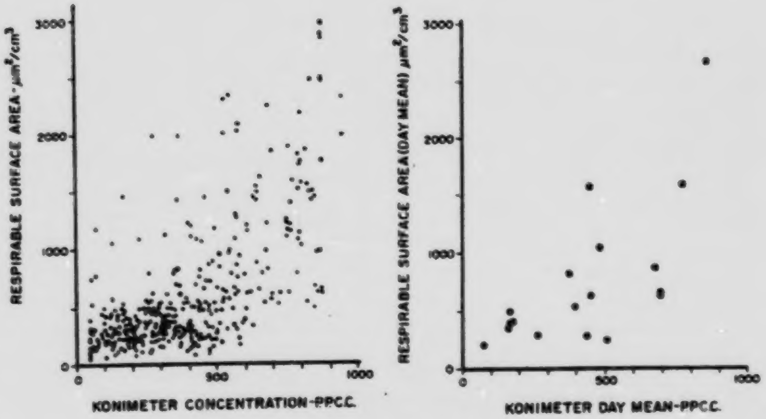


FIGURE 2A — Respirable surface area vs. number.

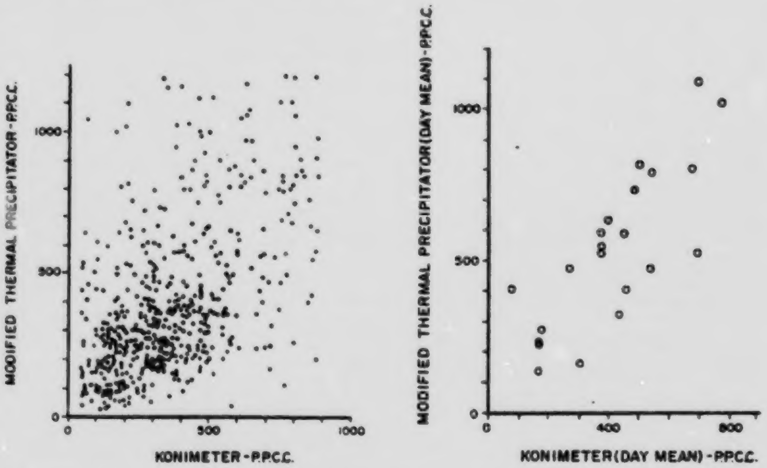


FIGURE 2B — Modified thermal precipitator vs. konimeter.

Findings and Observations on 1st-Year Studies, 1970

The following findings and conclusions may be drawn from the test work conducted in 1970 at six mines.

1. Sufficient encouragement was obtained of the potential value of fullshift gravimetric respirable dust sampling and quartz analysis for assessing exposure to warrant field testing these instruments in various mines in Ontario.

2. Excellent co-operation was provided by management, staff and workers at the mines where tests were conducted. Samplers were willingly worn by miners throughout working shifts, even though the harness to which the sampling equipment was attached was rather inconvenient to put on and take off. Very few samples were lost.

3. Concentrations of dust measured with short-period samplers varied widely, as illustrated by the scatter of plotted samples in Figures 2a and 2b. Konimeter readings (average of three spots) of samples taken at frequent intervals throughout shifts are compared with 5-minute DISA readings (Fig. 2a), which estimate respirable surface area (RSA), and also with 5-minute thermal precipitator (MTP) readings (Fig. 2b) from similar sampling periods. The scatter is less prominent when the day means are plotted in both instances. The samples were taken at three mines (A,B,C) having a high quartz content (60 to 70%) in the ores. The variations in dust concentrations during four operation cycles are plotted in Figure 3. Higher MTP readings compared with konimeter counts during drilling may be due to a greater collection and counting efficiency obtainable with the MTP when there are many very fine particles in the air.

Although the average of 26-day means was almost equal for the konimeter and the MTP, the range of the latter was greater, about 5:1 versus 3:1; this indicates some lack of linearity of the konimeter.

The variations indicated above indicate the difficulty of obtaining a time-weighted assessment of dust exposure with short-period samplers.

4. Readings taken with a short-period sampler (konimeter) and a full-shift gravimetric sampler are plotted as number versus mass. They also varied significantly for total respirable dust (Fig. 4a). This is true even though konimeter day means were plotted against day mass readings for high-, medium- and low quartz mines. The ore in Mine D contains about 8% quartz and Mines E and F about 20%. However, correlation is reasonably close when konimeter day means are plotted against respirable quartz readings (Fig. 4b) instead of total respirable dust.

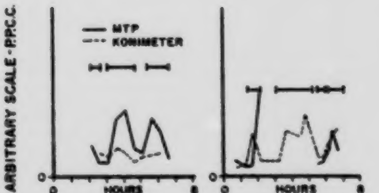


FIGURE 3 — Operating cycle variations.

5. The respirable surface area (DISA) readings from a limited number of MTP samples taken in both high-quartz and other mines did not reflect the increases in exposure hazard indicated by respirable-quartz mass measurements, as illustrated in Figure 5. The authors suspect that the DISA instrumentation, in its present state, does not give a true indication of surface area. Further research is required to confirm or alter this finding.

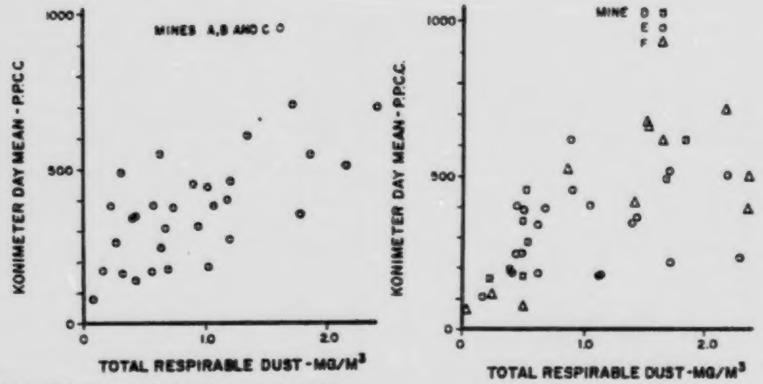


FIGURE 4A — Number vs. respirable mass.

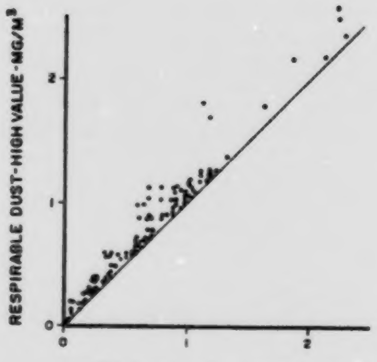


FIGURE 6A — Two fixed-position samplers in the same working place.

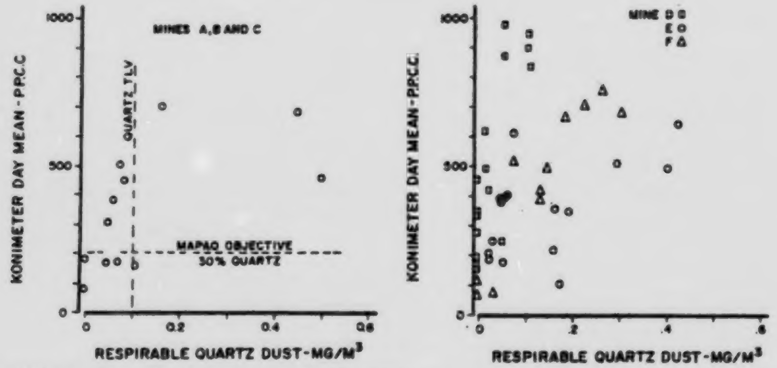


FIGURE 4B — Number vs. respirable quartz.

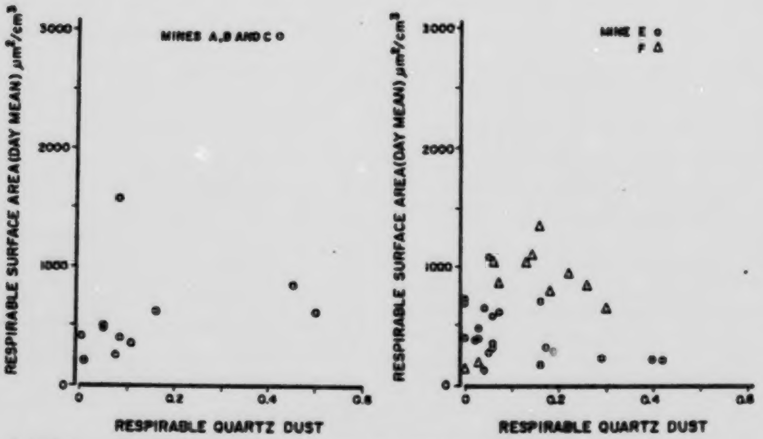


FIGURE 5 — Respirable surface area vs. respirable quartz.

TABLE 1 — Acceptability Rating*

(i.e. dust concentration less than standard)

ACGIH 0.1 mg/m³	MAPAO Objectives 200 ppcc	No. of Sites	300 ppcc	No. of Sites
Yes	Yes	12	Yes	18
No	No	29	No	28
Yes	No	20	No	14
No	Yes	1	Yes	2

*Yes, if dust concentration is less than standard; no, if greater than standard.

6. Table 1 presents an acceptability rating of comparative number, by konimeter, and mass readings with two-stage personal gravimetric samplers and quartz analysis at 62 working sites in high-quartz mines. At about 65% of the sites the two standards agree in either proving or disaproving them. An agreement of 75% was obtained if the former 300-ppcc objective for the konimeter was used. Many more comparisons would be required to confirm these relationships.

7. The data in Table 2 suggest the following comments.

(a) In stope drilling and slushing there is a reasonable correlation between the mean counts of short-period samplers (konimeter and MTP) and readings with full-shift gravimetric samplers.

(b) The difference between MTP 1 to 5μ and all visible counts indicate the significant number of particles below one micron.

(c) Konimeter readings for stopes are about 1¾ times the objective of 200 ppcc and three times the objective at the underground crusher and loading pocket. However, the respirable quartz is just under the objective (0.1 mg/m³) for stopes, but 7 and 16 times the objective at the crusher and loading pocket. Although this comparison is based on sampling only two and three shifts, respectively, at the crusher and loading pocket, it does suggest that the konimeter underestimates the hazard in high concentrations. This finding requires a more intensive investigation.

8. Figures 6a and 6b indicate similar concentrations of respirable dust for two adjacent fixed samplers and for two adjacent samplers worn by miners, respectively. There is a little difference in the latter,

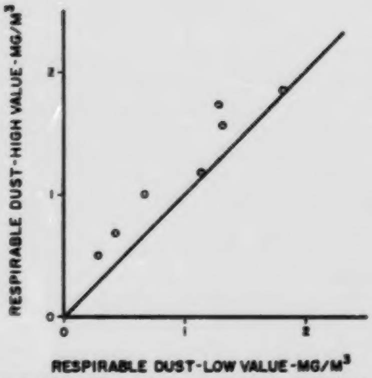


FIGURE 6B — Two personal samplers in the same working place.

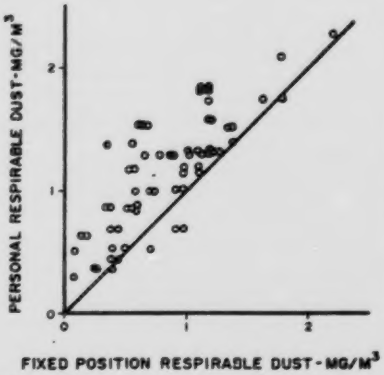


FIGURE 7 — Personal vs. fixed-position samplers.

TABLE 2 — Average Dust Concentrations at the Main Types of Mining Operation Examined

Instrument and Assessment Method	Average Dust Concentration			
	Stope Drilling *(14)	Stope Slushing *(13)	Underground Crusher *(2)	Skip Loading *(3)
Konimeter, ppcc	330	360	580	610
MTP 1-5 μm ppcc	200	150	305	456
All visible ppcc	400	540	460	1000
Total Surf. Area, μm²/cm³	1250	1450	1300	2990
Resp. Surf. Area, μm²/cm³	480	650	630	1950
Personal Samplers (fixed)				
Total resp., mg/m³	0.68	0.90	1.45	2.0
Resp. quartz, mg/m³	0.09	0.09	0.7	1.6

*number of shifts contributing to average.

TABLE 3 — Composition of the Respirable Dust

Site	Quartz % Mean	Quartz % Range min.	Quartz % Range max.	Combustibles % Mean	Combustibles % Range min.	Combustibles % Range max.
Mine A, B and C Working place	27.7	0	69			
Mine C only Working place	18.0	0	41	65	11	96
Mine D Working place	1.5	0	2.3			
Mine E Working place	6.5	0	20.6	48.2	5.4	97.5
Mine F Working place	6.4	0	12.8	55.5	36.3	70.4

Approximate amount of quartz in ore is 60-65% in Mines A, B, C, 8% in D and 20% in E and F.

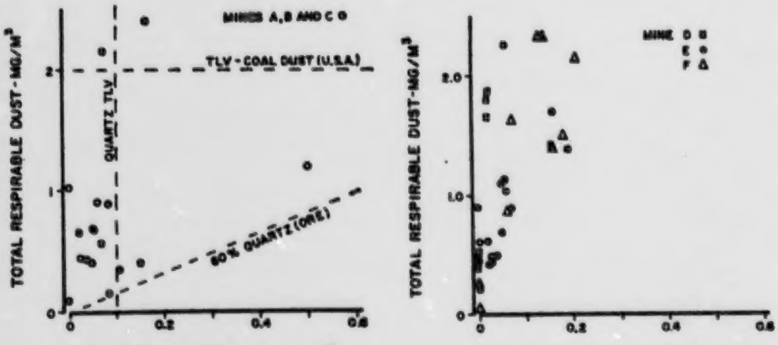


FIGURE 8 — Total respirable dust vs. respirable quartz.

probably because the miners move about. The high reading on any given day was plotted against the low reading. All samples were taken in high-quartz mines.

9. Daily readings from personal samplers worn by miners and those in fixed positions nearby are plotted against each other in Figure 7. The concentrations at the miners breathing zones are about 25 per cent higher than at fixed positions. This would indicate that samplers worn by workers will provide a more accurate assessment of exposure, but correction factors for use of fixed samplers at some operations could be established if necessary.

10. Figure 8 illustrates the wide variation of total respirable dust compared with respirable quartz. In three high-quartz mines most samples varied by a factor of 3 to 5, a few over 15. A similar relationship was found from readings taken at a mine with low quartz and two with medium amounts. The variation is probably due mainly to diesel exhaust particles.

This indicates the usefulness of personal samplers for measuring both total respirable dust and respirable quartz.

11. Table 3 indicates the proportion of quartz and combustibles in total respirable dust in the places sampled at the six Ontario mines. The respirable quartz amounts to between one-fifth and one-third of the total quartz in the various ores.

Field Testing Personal Gravimetric Samplers

Personal gravimetric samplers were field-tested at six mines in four mining areas. The tests were conducted by the technologist employed by the Foundation in cooperation with local staff. Subsequent studies of concentrations of diesel exhaust particles were conducted at several base metal mines and of quartz dust in a silica processing plant.

OBJECTIVES

The following objectives were set for field testing:

(1) to determine suitable procedures for issuing personal samplers at the beginning of a shift and returning them at the end of a shift;

(2) to determine the acceptance of these dust samplers by the miners and to resolve any difficulties encountered in using them on all types of jobs;

(3) to determine the feasibility of suspending the sampler near an operator at some dust-producing operations;

(4) to obtain some correlation between the gravimetric sampler and the konimeter in a greater variety of mines for the purpose of establishing permissible concentrations related to past experience in the incidence of silicosis;

(5) to investigate, in cooperation with ventilation personnel, the usefulness of gravimetric samplers for control purposes; and

(6) to test the quartz analysis technique on samples from additional mines.

PREPARATIONS

Arrangements were made with the managements of the mines concerned for the procedure to be followed; this included explaining the purpose of the study to supervisors and workers. The technologist assisted in this latter aspect. Participation of the ventilation staff of each mine and of the Association was encouraged.

The original harness for carrying the sampling equipment was so inconvenient that it was replaced by a leather shoulder strap joined back and front to the cap-lamp belt. The personal samplers used during the previous year were reconditioned.

TEST PROCEDURE

1. A supervisor's beat was chosen for the test by management during discussion with the technologist.

2. The technologist had a supervisor wear a personal sampler and accompanied him around the beat on the first shift to make him and the miners familiar with the function and purpose of the instrument. In most mines, a number of other supervisors also tried the sampler for a full shift. Ventilation staff was also involved wherever feasible.

3. On subsequent shifts, each of several miners wore a sampler until practically everyone, including haulage crews and service men, wore it once or more.

4. The technologist assisted the miners at the beginning of each shift, usually in the cap-lamp room, to put on the samplers. He also started the pumps and collected the instruments at the end of the shift. The airflow of the pump was checked before and after each shift; this could be done within 5 minutes.

5. During the shift, the technologist also wore a sampler and took konimeter samples at regular intervals as near as feasible to the sampler worn by a miner.

6. Gravimetric samples were prepared and processed, including X-ray diffraction, at the Mines Branch Laboratory.

Field Testing — Findings and Observations

Field tests at the mines were terminated in January 1972. Several months were required to complete the laboratory work and compile the data. The principal findings follow.

1. Most of the objectives were either wholly or partially achieved.

2. A suitable procedure was developed for issuing samplers to miners at the beginning of a shift and collecting them at the end of the shift. This will be simplified by modifications described later herein.

3. The willing acceptance on the part of miners and supervisors to wear personal samplers for full shifts was very gratifying. Possibly participation in development of an instrument for the better protection of their own health was a factor; also, management, staff and the technologist deliberately practiced good communications.

4. The samples collected at mines with various mineral contents in the ores provided much experience in refining the technique for determining approximately 0.1-mg fractions of quartz directly on silver membrane filters. An accuracy within 0.02 mg or 20 per cent of the TLV for quartz is estimated. It is important that this be achieved without relying on weighing for high-quartz mines. For example, the TLV for respirable dust(6) in mg/m^3 is: 10 divided by (% respirable quartz + 2) or 0.15 mg/m^3 of air if there is 63 per cent quartz in the respirable dust. It is not yet feasible to weigh this quantity accurately because of minor damage during the handling of filters.

Some interference was caused in X-ray diffraction analysis by the presence of micas and other minerals, but this was not a serious problem(4). It took more time because additional X-ray readings were required.

The authors and research sponsors are fully aware of the need to have the accuracy of the quartz analysis confirmed by analysts in other laboratories. Steps in this direction have been initiated and will be pursued to the extent deemed necessary(8).

5. If this accuracy is confirmed, then full-shift personal samplers can be used with confidence to collect a single sample on a filter for determining a time-weighted average exposure to:

(a) total respirable dust (and smoke) by weighing;

(b) quartz by X-ray diffraction;

(c) Diesel smoke by ashing and reweighing (or by other means);

(d) possibly other airborne respirable contaminants.

6. Personal samplers may be either worn by workers or suspended and operated in fixed positions nearby, in which case a correction factor should be established in each application.

7. Some breakage of plastic cases, housing the battery motor and pump assembly of samplers, was experienced when miners and supervisors were climbing in narrow manways. This problem will be eliminated in modifications described later.

8. The involvement of ventilation engineers and surveyors during the studies, in order to evaluate the usefulness of personal samplers for control purposes, was not as extensive as anticipated. However, their value was demonstrated in a few instances. Several poor practices in dust control, picked up by the full-shift personal sampler, were apparently missed by konimeter sampling. In one instance an assayer was brushing pulp from an open pulverizer toward his face.

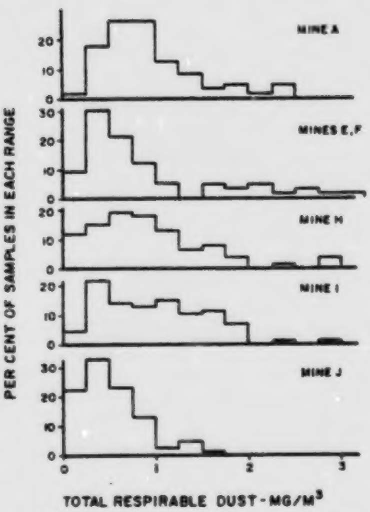


FIGURE 9A — Total respirable distribution.

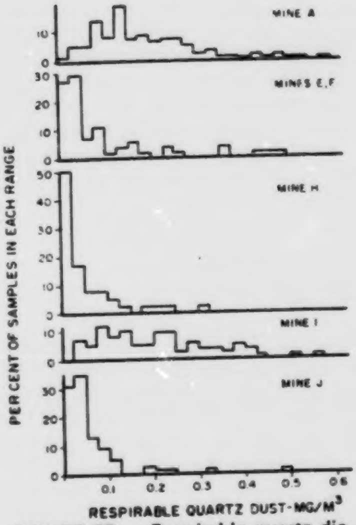


FIGURE 9B — Respirable quartz distribution.

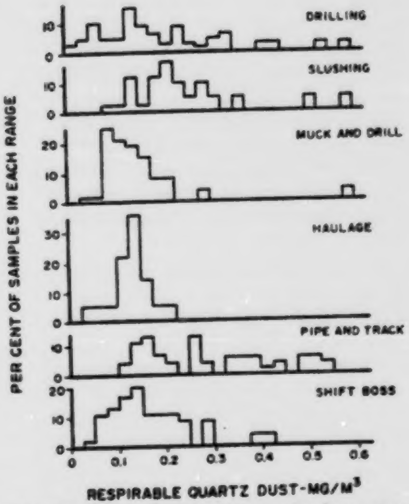


FIGURE 9C — Respirable quartz by occupations.

In another case auxiliary ventilation was inoperative. The personal gravimetric samplers in effect supplemented personal inspection.

The authors believe advantage can be taken of personal samplers for promoting more effective control because the workers and supervisors will be participating in the measurement of their own exposure and accepting more responsibility for their dust control practices. With a proper approach to workers and supervisors their participation and cooperation can be gained. This will be facilitated with modifications to the sampler described later herein.

Monitoring of the mine atmosphere should become an integral part of operations, as in checking ore grade in mining, and pH, reagents, etc. in mineral processing. Another example is the use of film badges and dosimeters in the nuclear industry. Also, full-shift noise dosimeters have been developed and put into use.

9. Several methods of transporting collected samples on filters have been tried. In the most recent approach the filter holder is shipped without disturbing the sample. The cyclone is removed from the holder and replaced by a plug having the same thread. Ten holders and plugs containing filters, made of magnesium, have been transported between the Elliot Lake Research Laboratory and a silica plant for about one year with no apparent disadvantages.

10. In order to prevent potential restrictions to airflow due to deposit of diesel smoke on filters, the Foundation had several 37-mm (instead of 25-mm) filter holders made of magnesium, thus almost doubling the effective filtering area. These were used on personal samplers for measuring the concentrations of combustible respirable particulates in the atmosphere at several base metal mines where diesels are extensively used. The samplers were operated for full shifts without significant reduction in airflow. The findings, reported elsewhere by Hews and Rutherford(10), are quoted in part below:

> "1. Using 25-mm-diameter filters, a total of 20 samples were obtained; average sampling period was 3 hours (all working time). Average results — total respirable dust = 2.9 mg/m^3; 56% combustible = 1.6 mg/m^3; rock dust = 1.3 mg/m^3.
>
> 2. Using 37-mm-diameter filters, a total of 15 samples were obtained, all 8-hour samples, from collar to collar. Average results — total respirable dust = 2.0 mg/m^3; 75% combustible = 1.5 mg/m^3; rock dust = 0.5 mg/m^3.
>
> 3. Using 37-mm filters, nine 8-hour samples were taken on slushermen in non-diesel areas to obtain figures on the background combustibles. Average results — total respirable dust = 0.9 mg/m^3; 50% combustible = 0.45 mg/m^3; rock dust = 0.45 mg/m^3.
>
> Thus the indications are that the respirable portion of the carbon particles in the air from our LHD engine exhausts are in the range of 1 to 1.5 mg/m^3."

The TLV for pure carbon black is 3.5 mg/m^3, and for coal mines in the U.S.A. it is 2.0 mg/m^3. The limit for British coal mines is 4.5 mg/m^3. The average content of quartz in the respirable dust is 1 to 3% in coal mines.

Little is known about the pathogenic implications due to the retention of two or more dusts in the human lungs. There is still less known about the changes, if any, which might occur from long-term exposure to diesel smoke underground, particularly when associated with other dusts.

The adverse experience in silicosis from exposure in coal mines followed by exposure to respirable quartz has been mentioned earlier. This suggests that some consideration should be given to the possible combined effects of long-term exposure to diesel smoke and over 5% of respirable quartz.

11. Distributions of concentrations are plotted as histograms — total respirable dust (Fig. 9a) and respirable quartz (Fig. 9b) at individual mines and at types of work at Mine A (Fig. 9c). They indicate that:

(a) most exposures in the places tested were less than 1 mg/m^3 of total respirable dust and 0.2 mg/m^3 of respirable quartz — during a few shifts concentrations were considerably higher;

(b) there were a few high exposures in most types of operations tested in one mine.

12. Gravimetric dust sampling was initiated in a plant processing almost 100 per cent quartzite about a year ago in the midst of an energetic program of dust control improvements. Since then, the portion of silica in the respirable dust dropped steadily from about 80 to 60 per cent. This is probably due to the greater friability of gangue material and also to the fact that more larger particles are airborne during dusty conditions compared with present acceptable concentra-

tions. Recently, respirable samples collected in the plant on Nuclepore, instead of silver membrane filters, were analysed for size distribution with a scanning electron microscope. The arithmetic and geometric mean sizes were 1.06 μm and 0.72 μm respectively. It is planned to obtain size analyses from samples taken at a high-quartz mine under similar conditions.

13. It is conventional at Ontario mines and probably elsewhere to take dust samples (e.g. with the konimeter) as close to the breathing zone of the exposed worker as feasible. Our studies confirmed the value of this practice.

14. In anticipation of incorporating most or all of the personal sampling components on the cap-lamp battery, a limited number of samples were taken with several miners wearing two units (rf. Table 4). The intake (cyclone and filter holder) of one was attached to the lapel as usual; the other filter holder was attached to the cap-lamp battery. Although the ratio between lapel and battery position readings varied considerably, the average (excluding samples with low mass and those taken under oilers) was 1.04:1, which was close enough to consider using the battery position regularly except in cases where clothing is worn over the battery. In rare instances when this is necessary the filter holder could be moved to the lapel area. Monitoring for an entire shift, with the filter holder attached to the lamp battery, would be a much more effective measure of the health hazard than three spot samples taken by a surveyor as near as practical to a miner.

Future Studies and Applications

The MAPAO has agreed to have its field engineers take over future field testing of personal gravimetric samplers in co-operation with various mining companies. Arrangements have been made to have some samples processed at the Mines Branch Laboratory, particularly the analysis of quartz by X-ray diffraction.

In anticipation of these studies the Foundation has financed the fabrication of six personal samplers with all components incorporated on cap-lamp batteries, as illustrated in Figure 10. This arrangement has several advantages.

1. The separate battery and charging facilities are eliminated.

2. A harness or shoulder strap is unnecessary because the filter holder and cyclone are mounted on the battery and a more compact pulsation damper is used.

3. There is less danger of the cyclone being tipped beyond a horizontal position and dislodging deposited dust from the filter.

The pump arrangement is similar to the Safety in Mines Research Establishment's Simpeds personal sampler described by Harris and Maguire(11), but the Simpeds has the cyclone and filter holder incorporated with the cap lamp, requiring a hose leading to the pump on the cap-lamp battery.

Two Simpeds samplers, recently manufactured by Casella, have been ordered for field testing. Some problems may be encountered from bumping and tipping of the cyclone and filter either during or at end of working shifts, which could result in spoiled samples.

Initial field testing arrangements have been made at high-, medium- and low-quartz mines. Some objectives of future field testing are as follows.

1. To test the usefulness of six modified and two Simpeds samplers.

2. To provide an opportunity for additional mines to try full-shift samplers and assess their usefulness for assessing exposure and for engineering control.

3. To compare readings from full-shift samplers with those from konimeter samples taken in the same working places during semi-annual surveys. Several years may be required to integrate full-shift samplers into semi-annual surveys.

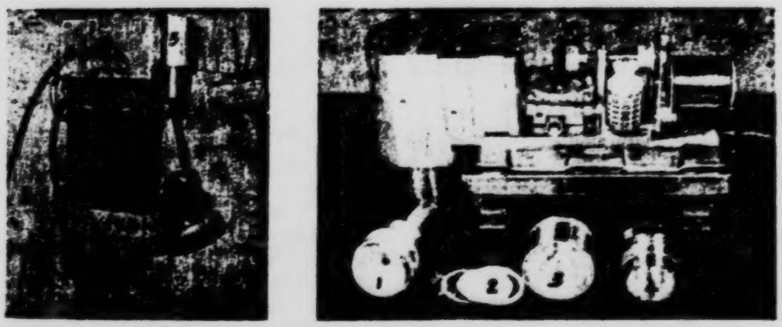
4. (a) To determine statistically the optimum frequency of operating full-shift samplers for assessing exposure;
(b) to investigate the advisability and feasibility of collecting a sample over more than one shift.

5. To obtain additional information regarding the concentrations of respirable quartz and of diesel exhaust particles in mine atmospheres.

TABLE 4 — Comparison of Lapel and Belt Sampling Positions

	Ratio of Belt to Lapel Dust Concentrations					
Drilling	0.7	1.1 1.0*	1.0* 0.5+	2.0* 0.25+	1.2	0.8*
Slushing	1.0	1.1	0.8	1.2		
Haulage	0.7	0.2*				
Muck and Clean-Up	1.0	1.6				

*Ratio inaccurate because sample weight was very low.
+Sampling head covered by protective clothing (oilers).



1. Cyclone
2. Filter
3. Filter holder
4. Plug (replaces cyclone for shipping)
5. Protective shield (for cyclone and filter holder)
6. Pulsation damper
7. Diaphragm pump
8. Minute counter
9. 4-volt motor
10. Removable cover
11. Air inlet

FIGURE 10 — Miner's Respirable Dust Sampler.

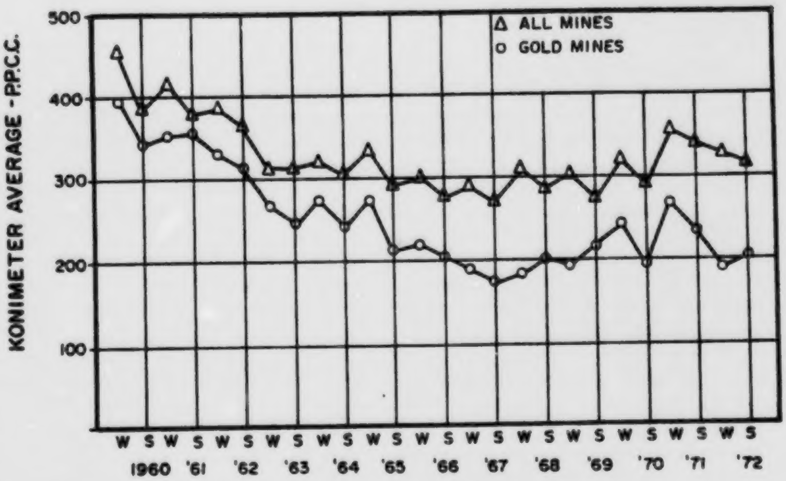


FIGURE 11 — Semi-annual dust surveys in Ontario mines.

6. To establish for certain occupations, if feasible, a reliable relationship between readings from full-shift samplers worn by a worker and those operated in fixed positions nearby.

7. To provide a greater variety of samples for refining the techniques and improving the accuracy of quartz analysis.

Conclusions

The following conclusions may be drawn from this research.

1. The personal two-stage full-shift gravimetric sampler has met the challenge that has been outstanding for many years. It is a useful and practical instrument for assessing the exposure of workers on a time-weighted basis.

2. It is not intended to replace the konimeter as a control instrument, but the personal sampler may be used for this purpose in some applications.

3. It can be used either as a personal sampler worn by a worker or operated nearby in a fixed position, in which case a correction factor should be established and applied.

4. The same sample can be used to determine: total respirable air contaminants by weighing; respirable quartz by X-ray diffraction; respirable diesel exhaust smoke by ashing and reweighing; and possibly other contaminants such as heavy metals. For the latter, larger samplers and several shifts of exposure may be required, or the bulking of samples in the laboratory.

5. Silver membrane filters are the most suitable because of insensitivity to moisture and static and their suitability for the direct determination of quartz by X-ray diffraction; that is, without weighing and without transferring the sample.

6. Further research, experience and cross-checking is required to confirm and improve the accuracy of analyzing small samples of respirable dust for about 0.1 milligram of quartz and other contaminants.

7. Participation of a variety of mines in studies with full-shift samplers is advocated, especially those mines handling ores with significant amounts of quartz.

8. The use of full-shift samplers for routine assessment of dust exposure would permit the ventilation engineer to devote more time to travel with supervisors. His abilities could therefore be better utilized to promote good dust control practices and more efficient use of ventilation facilities. What better way is there to integrate environmental control with production?

9. Equally important, the accumulation of data from the analysis of respirable mixed dusts, collected by full-shift sampling, will provide meaningful criteria for establishing safe concentrations of silica, diesel smoke and other contaminants.

Acknowledgments

The co-operation received from the management, supervision and workmen at the mines which participated in the field studies was most gratifying. A good deal of credit is due to the technologist, R. Kowalchuk, who conducted the field sampling, maintained the instruments and compiled most of the data. Many helpful suggestions were contributed by members of the Association's Subcommittee on Dust Control and Ventilation (P. Chmara, P. F. Pullen and J. G. Rutherford). The authors also wish to thank W. B. Dix, president, and the directors of the McIntyre Research Foundation for making this research possible and providing the opportunity to publish this paper.

APPENDIX A

The introduction of dust sampling and control in Ontario mines in the mid-twenties has been previously described(13). At the beginning of the last decade the MAPAO and the Ontario Mining Association convinced all mining companies in the Province that they should adopt more frequent dust surveys. Semi-annual surveys of dust exposure (using standard konimeter procedures) and ventilation in places where men work and travel underground and at surface operations were initiated in 1959. This practice had been previously adopted by uranium mining companies to help control radiation and highly siliceous (quartz) dust.

Following the adoption of regular dust surveys the average dust concentrations for all Ontario mines dropped sharply from around 460 to 300 ppcc between 1959 and 1965. Since then, the averages have more or less followed an even plateau (Fig. 11).

True incidence rates of silicosis among Ontario miners are difficult to obtain, because the composition of the group at risk has changed over the years and an ever-increasing percentage is being found among those who left dust years ago and in many cases are no longer in the industry. However, a very useful measure of the silicosis problem at any given time is provided by the number of new 5 ratings per 1000 mine employees in dust. A 5 chest rating is applied by the medical examiners to the first radiological sign of silicosis. Not all 5 ratings progress to a disabling condition and some of them never become "silicotic". The Medical Statistics Unit of the Workmen's Compensation Board records that the number of new "5s" has dropped from a peak of 6.6 per 1000 in 1931 to 1.3 in 1971. This encouraging decline is emphasized by a drop to a lower level in 1947-48. The general introduction of aluminum prophylaxis three years earlier at gold mines supplemented medical and engineering control measures.

Notwithstanding this encouraging trend, an average of 33 silicosis cases a year were reported as "found" by the Mines Accident Prevention Association in the five-year period 1967-71 (rf. Table 5). Most of these were first exposed to dust prior to 1930. In the gold mines only 9 cases are attributed to miners whose first dust exposure occurred after 1944 and, of these, seven are known to have had post-1944 coal mining exposure prior to entering gold mining in Ontario.

This complicating factor — the superimposing of quartzitic dust on earlier dust exposures — is also evident among the high-quartz uranium mines. Out of 42 uranium cases shown, 33 entered dust exposure in mining a year or more before the first uranium mine came into production in 1955. Many of these had previous gold or coal mining exposure. It is encouraging at least that no cases have been reported among uranium workers entering mining after 1960, when dust concentrations were partially reduced and other control measures were in effect.

The relationship between silicosis and dust surveys was not sufficient to provide a reliable indication of safe concentrations for specific types of ores or proportions of silica therein. Tentative objectives were proposed (500 ppcc for < 10% qtz., 300 ppcc for 10 to 30% qtz. and 200 ppcc for > 30% qtz.). Association officials have been for years fully aware of the need to measure the concentration of quartz and provided space on survey forms for this purpose. However, convenient means of determining the amount of quartz in respirable dust were not available. The indices used in research studies for German mines(13) by means of ore analysis was considered. Quartz was assigned a value of 1, feldspar 0.7, sericite 0.7 with < 25% qtz., 0.5 with > 25% qtz. and 0.3 with no qtz., coal and other ores 0.1 and calcite 0. Limited studies using a Casella Hexhlet sampler, which separates respirable from non-respirable airborne dust, were conducted at several Ontario mines. The studies indicated wide variations between the amount of quartz in the ore and in the respirable dust and also between different parts of a mine. This indicated that the German indices were unsuitable for Ontario mines. It was concluded that an instrument was required which would more accurately sample the silica-bearing dust cloud in relation to the risk involved. It should sample over an extended period and collect that portion of the dust capable of reaching the alveolar regions of the lungs. The sample should also be suitable for identification of the degree of potential hazards.

The Johannesburg(14) and Los Alamos(15) conferences at the beginning of the last decade stimulated extensive efforts to find better methods of assessing dust exposure. Walton(16) described the consensus of research scientists and medical authorities on basic principles as follows.

> **"First, the 'respirable' fraction of the cloud should be measured, that is to say, the small particles which can pass beyond the communicating airways of the lungs (which are provided with effective dust clearance mechanisms) and into the respiratory region. Secondly, the quantity of respirable dust is best measured by its mass (or possibly by surface area for quartz), perhaps with weighting factors according to the chemical or mineral composition. Thirdly, the hazard depends on the time-average concentration, conveniently measured over a shift rather than on the short-period maximum levels."**

In view of the conclusion among experts that surface reaction of quartz particles in the lung cells is responsible for fibrosis, experts in South Africa first concentrated on designing instrumentation for measuring surface area and produced the DISA instrument described by Talbot(6) and Beadle(17) and discussed earlier herein. Other methods are also being investigated.

In the United Kingdom, and somewhat later on this continent, gravimetric instruments were developed to collect and measure respirable fractions of dust and to analyze very small samples (< 1 mg) for quartz content. For this purpose, Walton(16) states that:

> **"The Medical Research Council Panels and the Johannesburg Conference recommended a selection curve for the selective sampling of respirable dust such that 50% of particles having a falling speed equal to that of a 5-μm-diameter sphere of unit density (5 μm UDS) and no particles greater than 7.1 μm UDS are collected. This form of selection can be obtained quite simply by drawing the cloud through a 'horizontal elutriator' comprising a number of shallow rectangular-section ducts formed between a number of flat metal plates stacked one above the other."**

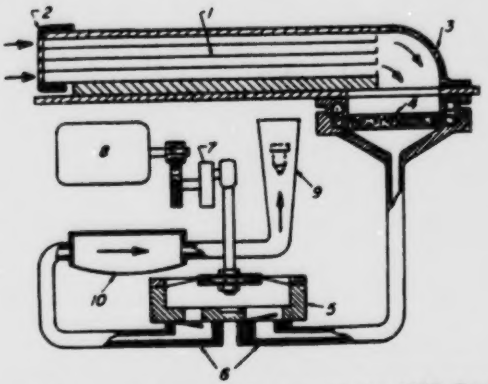
The first instrument developed to meet these specifications was the Casella Hexhlet sampler described by Wright(18) and used extensively in British foundries and coal mines. By means of a compressed-air ejector, air, at a rate of 50 lpm (litres per minute), is drawn through a horizontal elutriator in which coarse dust settles, leaving the respirable particles to be collected on a filter. It is most useful for collecting respirable dust for analysis, as mentioned earlier.

The Mining Research Establishment (MRE) of the National Coal Board (NCB) in the United Kingdom then developed a portable battery-driven instrument with selective characteristics similar to the Hexhlet, but using an air flow of only 2.5 lpm. The principal of operation and the MRE Sampler are described by Dunmore *et al.*(19) and illustrated in Figure 12.

TABLE 5 — Review of Silicosis Cases, 1967-1971

Period First Expos.	Before 1967	Year Found 67	68	69	70	71	Group Totals G	O	U	S	Total
Before 1930	1,445	26	21	18	20	12	1,387	155	—	—	1,542
1930-34	49	2	4	1	7	2	54	9	1	1	65
1935-39	19	4	4	2	—	4	24	5	2	2	33
1940-44	6	1	2	1	4	2	6	3	6	1	16
1945-49	8	3	3	3	2	3	7	2	13	—	22
1950-54	4	2	3	3	1	2	2	1	11	1	15
1955-59	6	—	2	—	—	1	—	—	9	1	10
1960-64	1	—	—	—	—	—	—	—	—	1	1
1965-69	0	—	—	—	—	—	—	—	—	—	—
G	1,365	26	27	22	22	18	1,480				
O	152	7	3	1	9	3		175			
U	16	4	9	6	2	5			42		
S	5	1	—	—	1	—				7	
TOTAL	1,538	38	39	29	34	26					1,704

Note: G — Gold
O — Other Than Gold, Uranium & Silica
U — Uranium (exposure in uranium mines after 1954)
S — Silica
From MAPAO 41st Annual Report, 1972



1. Horizontal elutriator
2. Inlet guide
3. Removable elbow
4. Filter
5. Diaphragm pump
6. Valves
7. Adjustable crank
8. Motor
9. Rotameter
10. Pulsation damper

FIGURE 12 — Schematic diagram of the MRE Sampler.

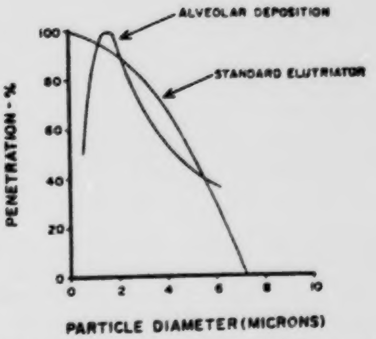


FIGURE 13 — Standard elutriator cut-off and alveolar deposition.

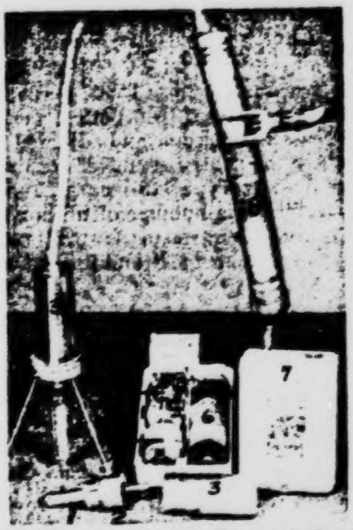


FIGURE 14 — Casella Personal Gravimetric Sampler.
1. Cyclone
2. Filter holder
3. Protective shield
4. Diaphragm pump
5. Motor
6. Ni-Cd battery
7. Plastic case
*8. Pulsation damper
9. Flowmeter

*Since replaced by compact damper.

The relationship between the horizontal elutriator cut-off compared with alveolar deposition is illustrated by curves in Figure 13 from studies described by Davies (30). The MRE Sampler (Casella Type 113A) has been used as a standard for respirable dust sampling by the NCB and the U.S. Bureau of Mines (USBM) for some years. Though it is suitable for whole-shift operation either in fixed positions or carried by a surveyor, it is not feasible for use as a personal sampler.

In order to meet this requirement, the British Cast Iron Research Association (BCIRA) developed a personal gravimetric sampler using a metal cyclone to separate respirable from total airborne dust as described by Higgins and Dewell(31). The close match in selection of dust to the MRE Sampler is achieved by using a flow of about 1.9 lpm. A commercial unit (Casella Type 13040) is shown as worn by a miner in Figure 1; Figure 14 is a photograph of components in a test arrangement.

In the United States, several types of cyclones were researched, but the 10-mm-diameter nylon cyclone was adopted for routine use in coal mines on the basis of studies reported by Jacobson and Tomb(32). It was also used in comparative work with the midget impinger in Vermont granite sheds and some foundries for the purpose of establishing a TLV for quartz on a mass basis as described by Ayer, Sutton and David(33), and subsequently by Ayer(34). These were very important developments. The first stage, either the horizontal elutriator or the cyclone, fulfills the function of the nasal and bronchial passages in trapping coarse dust particles (rf. Fig. 13). The second stage, an efficient filter, catches the fine particles which would normally penetrate the alveoli (air sacs) of the lungs and potentially cause fibrosis.

In the past ten years, a substantial amount of enlightening information on the engineering and medical aspects of dust exposure was made available at annual American Industrial Hygiene conferences and an occasional symposium sponsored by the McIntyre Research Foundation. This information, the need for qualitative assessment of dust outlined above, and progress achieved in other countries in collecting and analyzing small (< 1 mg) samples for quartz prompted some action in Ontario under Canadian conditions.

The MAPAO Subcommittee on Dust Control and Ventilation set the following objectives: (a) development of simple instrumentation to collect respirable samples for weighing and for occasional analysis of quartz content; (b) full-shift monitoring in order to achieve a time-weighted average measure of dust exposure; (c) laboratory facilities for analyzing samples for quartz down to a weight of < 1 mg instead of the several hundred currently required by the phosphoric acid method; (d) correlation of mass and number of particles, which would require many parallel samples.

Efforts toward achievement of several of the objectives were expedited by a visit of Canadian representatives from the federal and provincial mines and health departments and a mining company to research laboratories of the U.S. Public Health Service in Cincinnati and the Bureau of Mines in Pittsburgh. Subsequently, one of the Canadian representatives, Dr. J. Leroux(4), developed and published a procedure for direct determination of quartz on silver membrane filters which proved to be a very important key in the success of research described in this paper.

In the meantime, gravimetric sampling and weighing of respirable siliceous dust had been initiated at Rio Algom's Nordic mine at Elliot Lake. An arrangement for sampling both total airborne and respirable dust was assembled and tested. Weighing was done on a micro-balance at the Mines Branch's Elliot Lake Research Laboratory and some samples were analyzed for quartz by Dr. Leroux at the Environmental Health Centre, Department of National Health and Welfare, Ottawa. Encouraging results reported by Pullen and Yourt(35) convinced the Association's Committee on Occupational Health and its directors to expedite this research. Consequently, in 1969 the McIntyre Research Foundation was invited to conduct and finance intensive research on dust sampling.

APPENDIX B

Instruments Tested

The following dust samplers were purchased for testing their usefulness.

1. Three Casella Type 113A samplers fitted with horizontal elutriators to meet BMRC (British Medical Research Council) specifications(16). This type is most suitable for use in fixed positions and as a standard instrument. Respirable dust is collected on a 55-mm-diameter filter.

2. Ten Casella Type 13040 personal samplers, each fitted with a metal cyclone size selector and adjusted to collect the same amount of respirable dust as Type 113A on a 25-mm filter(21). This number of units was required to operate several samplers simultaneously and for spares.

3. One MSA gravimetric dust sampling kit.

4. One Unico Micronair personal sampler.
The MSA and Unico samplers, used extensively for sampling coal dust in the U.S.A., are fitted with 10-mm nylon cyclones calibrated to ACGIH specifications(26). Preliminary tests indicated low readings with siliceous dust, possibly due to the lack of an effective pulsation damper. This deficiency has since been corrected.

5. One South African Modified Thermal Precipitator (MTP). This unit consists of a battery-driven pump and hot wire and takes fifteen 5-minute 50-ml samples on a standard microscope slide.

In addition to the instruments listed above, a konimeter and a Casella Long-Period Dust Sampler (LPDS), both available in the Laboratory, were used for comparative studies; the LPDS is a thermal precipitator modified to run for a whole shift.

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(Reprinted from The Canadian Mining and Metallurgical Bulletin, April, 1974)
Printed in Canada

1. 4

Section 1—Chapter 4

Development of a Dust Sampling System for Hardrock Mines Based on Gravimetric and Quartz Assessment

by

G. Knight, R. Kowalchuk and R. Yourt

American Industrial Hygiene Association Journal
35(11) pp671-680 (1974)

MISE AU POINT D'UN SYSTÈME D'ÉCHANTILLONNAGE DE LA POUSSIÈRE BASÉ SUR DES MESURES GRAVIMÉTRIQUES ET SUR UNE ÉVALUATION DE LA CONCENTRATION DE QUARTZ POUR LES MINES DE ROCHE DURE

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Dans le présent rapport, on analyse les résultats d'échantillonnage de la poussière obtenus au laboratoire et sur le terrain. On a trouvé qu'il était possible d'utiliser des échantillonneurs-classificateurs de poussière gravimétriques (petits appareils alimentés par une batterie) individuels ou fixes, équipés de filtres à membrane en argent, et d'évaluer les concentrations de poussière inhalable en termes de poussière totale, proportion de quartz (par diffraction directe des rayons X sur l'échantillon) et proportion de poussière combustible (après incinération). On a constaté que la concentration et la composition de la poussière étaient très variables et l'échantillonnage doit se faire sur plusieurs postes de travail pour que l'on obtienne une estimation raisonnable de l'exposition à la poussière. En raison de cette grande variabilité, il est nécessaire de concevoir un programme d'échantillonnage en vue d'une analyse statistique afin de réduire les coûts au minimum et d'obtenir le maximum d'information.

Development of a Dust Sampling System for Hardrock Mines Based on Gravimetric and Quartz Assessment

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This report analyses the dust sampling results obtained in laboratory and field studies. It was found possible to use fixed position or personal size-selecting gravimetric dust samplers (small battery-powered units) with silver membrane filters and to assess the respirable dust concentrations in terms of total dust, the quartz portion (by direct X-ray diffraction on the sample) and the combustible portion (after ashing). Both dust concentration and dust composition are found to be very variable and a number of shifts need to be sampled to give a reasonable estimate of the dust exposure. Because of this high variability, statistical design and analysis of a sampling program are necessary to minimize costs and maximize information.

Introduction

THIS PROJECT CONTRIBUTES to the Department of Energy, Mines and Resources objective of improving the working conditions in mines and in large part has been undertaken in cooperation with the McIntyre Research Foundation and the Mines Accident Prevention Association of Ontario.

The techniques presently used in the Canadian provinces were adopted from other countries either because there was known to be a silicosis problem, as in the Ontario gold mines, or to avoid the development of a problem. In many cases, these techniques were applied as spot checks and for engineering control and gave only a limited estimate of the miners' total exposure to dust. With the industry as it is today with dust exposures mostly under reasonable control, it is useful to develop a dust sampling system for possible routine use that gives an acceptable assessment of the health hazard, while at the same time being relatively easy to use and of some value for engineering control.

Many researchers have examined the correlation between different types of instruments in mines and laboratories and have shown that the correlation is poor. A study in this laboratory[1] on a range of pure mineral dusts prepared by different methods to give a range of particle size distributions, showed that, although consistent ratios between the indications of each type of instrument could be obtained in any one type of dust cloud, the ratios varied widely (4:1 to 40:1) between the different types of dust cloud. Presumably, therefore, there is an equally large variation in the ratio of the dust indicated by most samplers to the health hazard in different types of dust cloud, and therefore the relationship between dust sampler indication and the health hazard needs consideration.

Particle Size

Dust penetration to and deposition in the alveolar region of the lungs is for the most part dependent on particle size. This was first allowed for by rejection of coarse particles in microscope assessment of the dust. Two more recent approaches are: (a) a two-stage sampler simulating physically the deposition of dust in the two main regions of the lungs with the second stage of the sampler collecting the so-called 'respirable' dust which can form a long term health hazard when deposited in the alveolar region of the lung;[2-6] and (b) an optical method of dust sizing as exemplified by the South African technique DISA.[7]

The dust sampling system described in this report uses the first approach.

Composition

Free silica dust and asbestos fibres are generally much more harmful than other types of dust, and the permitted dust concentration depends greatly on the proportions of these dusts present.

Of these harmful dusts, quartz is the one most commonly found in mines, so as sensitive analysis techniques have been reported,[8] a technique has been adapted for easy use on mine samples.

Physical Parameter Assessed

The choice of parameters can be very important; for instance, particle counting accentuates the hazard of a dust cloud consisting of small particles whilst mass determination accentuates the hazard of a cloud with large particles.

Recently it has been shown[9] that the health hazard of coal mine dust is closely related to the mass in the respirable size range. It is widely assumed that mass is also the best measure for other dusts which do not give rise to a specific biological reaction. For silica the position is not clear and it is assumed, on the basis of the theories of the physiological action of silica, that the surface area of the silica dust in the respirable size range is most closely related to its health hazard.[2] However, there are no methods as yet acceptable which could be applied to mine dusts to measure specifically the surface area of the silica dust present. The available analytical techniques for quartz, such as x-ray diffraction and infrared absorption, measure parameters that are more closely associated with mass than with surface area. The authors conclude that these are the best estimates available now of the health hazard due to silica in mine dusts.

In support of this conclusion, the following evidence is presented:

(1) Goldstein and Webster's[10] conclusion, from intratracheal injection of sized quartz particles, that mass may be a better indicator of the silicogenic effect than surface area;

(2) W. H. Walton[11] said, "surface area (of dust particles) is not remote from mass, but number is, on the other hand, very remote" (from surface area); and

(3) Page-Shipp and Harris[12] concluded that there is a high correlation between respirable surface area and respirable mass of airborne dust in South African gold mines as determined by microscopy on thermal precipitator slides.

Because of this doubt and the uncertainty of the effects of other dusts mixed with silica, analyses of mine dust should be made in more detail on a small number of samplers. A suitable start to this might be to collect routine gravimetric respirable dust samples, bulking those from each job in each mine or section and analyzing them to obtain at least the following extra information: size distribution of all the respirable dust, size distribution of the respirable quartz dust, the major mineral components, the minor elemental components, and the major organic constituents.

Sampling Period

The onset of pneumoconiosis is related more to the total amount of dust inhaled than to the peak concentration during a shift.[2,9,13] A long period sampler makes a better estimate of this than does a snap (grab) sampler, so a full shift is usually considered the best compromise between accuracy and convenience and is the time period used in this study. However, an argument has been made for longer periods, about 10 days,[14] so consideration should be given to longer-period dust samplers in the future.

Personal or Working Place Sampling

For estimation of the miners' dust exposures, personal sampling is preferable, while working place sampling may be more useful for dust control and may lead to considerable economies in sampling if a considerable number of men are employed at one place, as for instance on a longwall coal face.[15]

This study describes the experimental work carried out underground and the related work undertaken in the laboratory on the testing and development of a dust sampling system in hard rock mines based on the above approaches. Full details of these studies have been reported in Mining Research Center internal reports (limited circulation) and a paper emphasizing other aspects of this study has been given elsewhere.[16]

Experimental Techniques

Size-Selecting Gravimetric Samplers

Four makes of size-selecting gravimetric samplers have been examined:

(1) Casella type 113A,[7] fitted with a horizontal elutriator size selector to MRC specifications,[3] using a 55-mm-diameter filter;

(2) Casella personal sampler type 13040,[7] fitted with a metal cyclone size selector which was adjusted to collect the same amount of respirable dust as (a) above on a 25-mm-diameter filler;

(3) and (4) The Unico[8] and MSA[9] personal gravimertic samplers used the same nylon cyclone size selector calibrated to the ACGIH[5] specification, and collected the dust on 37-mm-diameter filters.

Mass assessment was done by weighing the filters and x-ray diffraction techniques described in detail elsewhere[20,21] were used for determination of quartz. The quartz determination is most sensitively done on dust collected on a 10 x 20-mm silver membrane filter, *i.e.*, the shape that fits the x-ray beam. However, such a small area restricts the airflow and does not permit the collection of a weighable quantity of dust. The Casella personal gravimetric sampler using a 25-mm-diameter, 5-μm pore size filter (b, above) was found to give the most sensitive estimate of quartz and mass and had most use in the study both as a personal and as a fixed-position sampler. These samples can also be readily ashed at 500°C because the change in weight of the silver filter is limited to ±0.02 mg. The results are expressed as milligrams per cubic metre (mg/m^3) of total respirable dust, respirable quartz dust, or respirable combustible dust (loss in weight on incineration).

The Short-Period Samplers

Two short-period samplers were used for characterization of variations in dust concentrations and for comparison with the proposed full-shift sampling method. The konimeter, used in Ontario,[22,23] collects dust from 5-cm^3 samples of air on a glass slide by impaction. The slides are treated by incineration and acid wash (to emphasize quartz content) and counted by a scanner.[24] The South African modified thermal precipitator,[25] referred to as the MTP, takes a 50-ml air sample in five minutes and deposits the dust on a glass slide for counting under the microscope. Dust concentrations are expressed in particles per cubic centimetre (p/cm^3).

Calibrations

1. Flow Rates

Either sample volumes or flow rates of all instruments were checked periodically, on either volumetric glassware or wet test gas meter as a primary standard and taper tube flow meters as secondary standards. The manufacturer's recommendation[17] for the Casella personal respirable dust sampler (type 13040) states that the flow rate should be adjusted two or more times daily by bleed screw on the pump; however, in dusty environments, this can permit dust to enter the pump and clog the valves. For our purposes, good results were obtained by setting the diaphragm stroke to give 2 liters/min with the bleed screw closed. The flow rate then dropped slowly during the shift as the dust built up on the filter. The nomal drop in the shift was 0.1 to 0.2 liter/min. Sampling time was limited to 3 to 5 hours on some occasions by fine black particles apparently associated with diesel engine use.

2. Dust Collection Characteristics

The calibrations were done in the laboratory dust chamber using a coal dust cloud containing about 40% respirable dust as indicated by the Casella dust samplers. This type of dust cloud has previously shown the largest difference between size selectors.[1]

It was found that the respirable dust concentrations by mass indicated by all the size-selective dust samplers manufactured by Casella were within ±7% of the mean while those indicated by the American instruments were about half of this.

Underground Sampling Positions

A number of sites were examined during the course of this study. At each of these sites, attempts were made to take dust samples, (a) in the men's breathing zones, (b) at fixed positions downwind and as near as practicable to the miners, (c) in the air entering, and (d) in the air leaving the operation. The full-shift dust sampling instruments took continuous samples, whereas the short-period samplers were operated at regular intervals of 15 or 30 minutes.

Testing was undertaken in eight mines and a number of preparation plants. The ore in the mines examined had quartz contents between zero and 70%; and for the purposes of this report, the mines are divided by quartz content of the ore into three classes—high (over 30%), medium (10-30%), and low (under 10%).

Analysis of Dust Measurements

The analysis can conveniently be divided into a number of aspects: (a) comparisons between the measurements obtained by each type of instrument, (b) variations of dust composition, (c) variations of dust concentration with position, (d) variations of dust concentration with time, and (e) the pattern of dust levels shown by shift-period gravimetric samplers. These are examined in the following five sections. It should be noted that special efforts were made to take dust measurements in high concentrations to extend the range of comparisons and the concentrations given here should not be interpreted as representative of those to which the miners are exposed.

Konimeter Respirable Gravimetric Sampler Comparisons

A comparison between the day mean concentration given by the konimeter and the total respirable mass given by the gravimetric samplers is shown in Figure 1 for mines classified according to quartz content of the ore and for comparison measurements

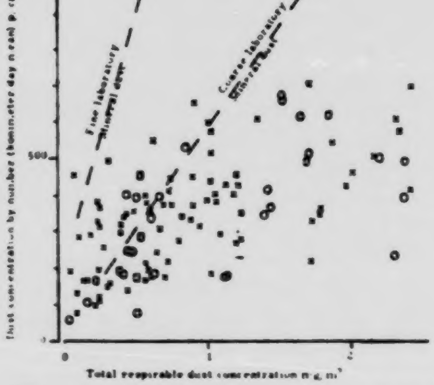

Figure 1. Comparison of Konimeter and respirable dust concentrations.

made on pure mineral dust in the laboratory chamber.[1] It can be seen that there is a very wide spread and that a mean ratio between the two assessments would be of very little value even in a mine that had uniform ore. In comparison to the laboratory tests, the results in mines show low konimeter readings at high mass dust concentrations, presumably due to two factors, the contribution of combustible dust to the gravimetric samples and the influence of averaging readings above 1000 on the konimeter as 1000 in deriving the day mean on a widely varying dust concentration.

However, the konimeter samples are heat treated and acid washed to emphasize the free silica content of the dust. The comparison with respirable quartz dust is shown in Figure 2. Though this still shows a wide scatter, a better agreement can be seen with the laboratory results on pure mineral dust for the high-quartz (65% in ore) mines.

Variations in Composition of the Dust

Two measurements were made of composition during the course of this study. Respirable quartz dust was assessed by x-ray diffraction and respirable combustible dust was assessed by ashing samples taken by the Casella cyclone gravimetric sampler. The composition of the respirable portion of the dust in selected samples from three mines is given in Table I with means and standard deviations. It is apparent that the proportions of combustible and quartz in

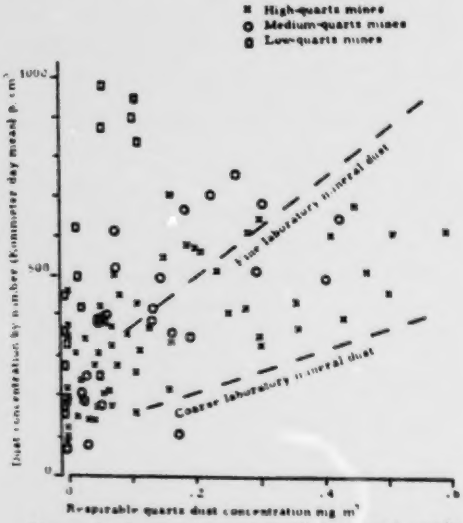

Figure 2. Comparison of Konimeter and respirable quartz dust concentrations.

TABLE I
Composition of the Respirable Portion of the Airborne Dust in Three Mines*

Combustible Dust %	Ash %	Quartz as % of Total Dust	Quartz as % of Ash
High quartz mine			
20	80	40	50
37	63	31	50
57	43	20	46
47	53	34	69
30	70	38	54
34	66	46	70
8	92	70	76
10	90	61	68
16	84	32	38
31	69	43	62
66	34	18	53
52	48	24	50
38	62	42	68
96	4	T	—
Range 8 to 96	9 to 92	0 to 70	0 to 100
Mean 57	43	22	52
Medium Quartz mine			
29	71	8	11
49	51	7	14
12	88	9	10
53	47	6	14
93	7	1	14
36	64	10	16
62	38	7	18
35	65	11	17
Range 5 to 100	0 to 95	0 to 15	0 to 33
Mean 49	51	7.5	15
Low Quartz mine			
24	76	2	3
23	77	2	3
96	4	T	—
54	46	2	4
77	23	2	10
Range 19 to 96	6 to 81	0 to 2	0 to 30
Mean 50	50	1.5	3

*Determinations selected as probably accurate, with ranges and means for all determinations.

the respirable dust are very variable; this is presumably due to the lack of correlation between their sources, mainly diesel engine exhaust and rock working, respectively. Thus it can be expected that the mass of total respirable dust would be a poor estimate of the mineral and quartz dust present. However, there is a fair correlation between the quantities of quartz and ash present.

Variations of Dust Concentration with Position

The results obtained can be considered in two parts: variations between intake, working place, and return and variations within the working place. The differences between intake, working place, and return depend on the ventilation and on the position and quantity of dust produced. For instance, the slushermen at the lower end of the stope were in either the intake or the return, depending on the direction of ventilation. The mean dust concentration at the intake, working place, and return, as indicated by fixed respirable gravimetric samplers is shown in Table II for stoping operations.

It can be seen that the dust concentration in the working place is generally higher than in the air entering the working place for drilling operations; however, this appears to be a very local increase due to poor dilution because it is not appreciably higher in the mixed air leaving the working place. Slushing produces a considerable increase in dust in the return as compared to the intake.

TABLE II

Mean Dust Concentrations at the Intake, the Working Place, and the Return Air in Stoping Operations at High-Quartz Mines

	Mean Respirable Dust Concentrations (mg/m^3)		
Operation	Intake	Working Place	Return
Jack-leg drilling	0.395	0.538	0.39
Long-hole drilling	1.23	1.33	1.19
Slushing upcast	—	0.53	0.93
Slushing downcast	0.12	0.63	—

Comparison between fixed position dust samplers in the same working place showed that in most cases the differences between them were not significant. The exceptions were apparently due to real differences in dust concentrations over a short distance. For example, in downcast slushing operations, the results suggested that channelling of dust in the airstream was frequent even when averaged over a period as long as a shift.

On about sixty shifts, personal gravimetric samples were taken on two or more men in the same working place. The ratio of the respirable quartz dust concentration for the high man to that for the low man ranged from 1:1 to 4:1. Also the value for personal samplers was usually greater—on the average 25% more—than for fixed-position samplers in the same working place. This is believed to be due to real differences in dust exposure rather than to greater experimental error when the samplers are worn and suggests that the local dust concentration around the man is increased significantly by his work and that personal samplers should give a significantly better estimate of his dust exposure than fixed-position samplers.

Variations of Dust Concentration with Time

The importance of the variability with time is the guidance that can be obtained relating to both the optimum sampling period and the frequency of sampling. These variations are conveniently considered in three aspects: (a) dust concentration variations during the shift, (b) shift-to-shift variations for one man, and (c) shift-to-shift variation in one working place.

1. Dust Concentration Variations during the Shift

Figure 3 shows the wide range of pattern for one type of operation, drilling, that is measured with short-period samplers—konimeter and MTP. The results for other types of operations are similar. It is apparent that, in some cases, the dust concentration varies only slightly with time but in others it varies precipitously. These variations sometimes, but not always, show a dependence on the periods of active work (as distinct from preparation and ancillary operations).

2. *Shift-to-Shift Variations in Dust for One Man*

Twenty-three men were observed for 3 to 6 shifts each, during the course of this study. It was found that the variability in quartz dust exposure for most men was such that the ratio of the maximum to the minimum shift mean quartz dust concentration was between 2 and 4. For only two men, could it be considered almost non-variable while five had ratios between 6 and an extreme of 37.

3. *Shift-to-Shift Variations in Dust for One Working Place*

The shift-to-shift variations for one working place are somewhat greater than the shift-to-shift variations for one man. This may reflect changes in the operation, however, and in its resulting dust level.

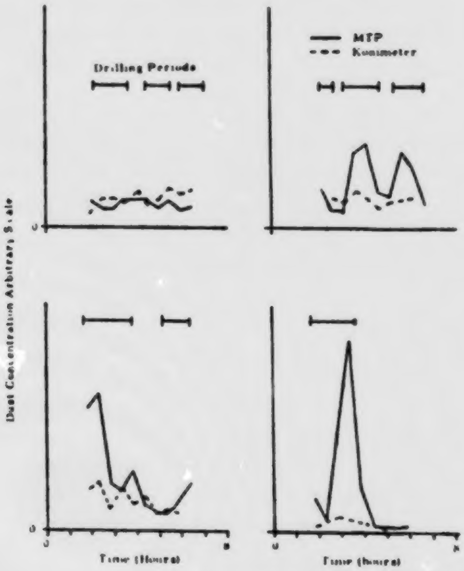

Figure 3. Range of patterns of dust concentration variations found during drilling shifts.

The Pattern of Dust Levels Shown by Shift Period Gravimetric Samplers

The results of a study in which respirable dust from sections of six mines widely spread over Ontario were sampled by personal gravimetric samplers were examined. These six mines were medium and high as to quartz content of the ore and, because quartz is the main hazard, the discussion is limited to the respirable quartz dust concentrations.

Figure 4 shows histograms of the frequency with which dust concentrations were found in each mine. It can be seen that a limited number of shifts are worked in concentrations considerably higher than the mean. Figure 5 shows histograms of the frequency with which dust concentrations were found for a number of occupations in one mine. Again it can be seen that the

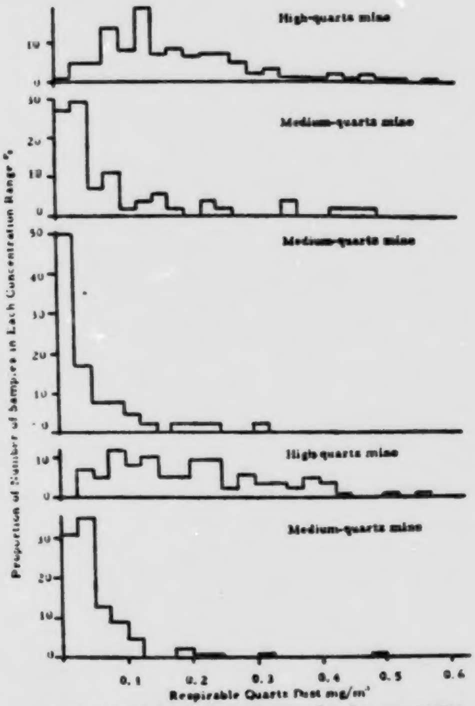

Figure 4. Distribution of samples by concentration range of respirable quartz dust.

occasional shift is worked in a high concentration of dust in most occupations.

Discussion

It has been shown that it is possible to collect dust on filters underground with small self-contained size selecting dust samplers—gravimetric samplers—and assess these in terms of the mass of total respirable dust, the mass of respirable quartz dust (by x-ray diffraction analysis) and the mass of respirable combustible dust (after ignition). The sensitivities of these three assessments on air samples of about one cubic meter are adequate, at about one-tenth of the threshold limit values being applied in related fields, *i.e.*, respirable quartz (0.1 mg/m^3),[5] applicable to dust containing more than 5 or 10% quartz; and coal mine dust (2 to 4 mg/m^3),[5] presumably applicable to mine dust containing less than 5% quartz or to combustible mine dust.

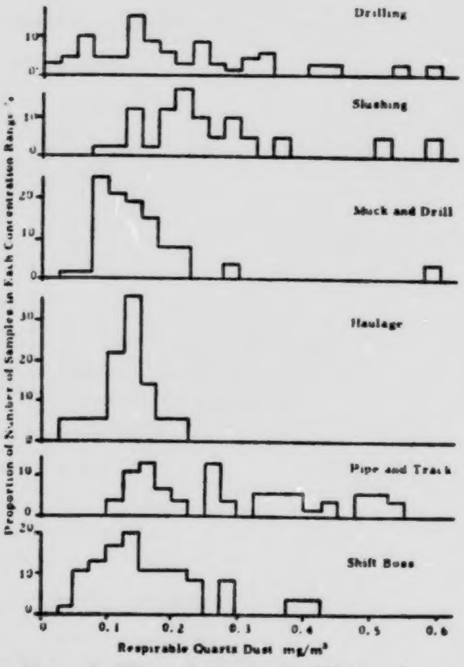

Figure 5. Effect of job on distrbiution of samples with concentration in a high-quartz mine.

Determination of Quartz in Mine Dust

The technique used has been described in previous papers.[20,21] Briefly, the x-ray intensities measured directly on the mine samples are compared with those from a previously calibrated permanent quartz sample to give a quantitative estimate of quartz, which is then corrected for the thin deposit loss as described previously.[20] The main problem found is interference by other minerals; however, four diffraction lines of quartz can be used for quantitative analysis and, so far, one or more of these lines has always been found to be clear of interference.[21] In some mines, the interference varies from sample to sample and it is necessary to cross-check using two or more quartz diffraction lines.[21] The lower limit for assessment of quartz is set more by identification of diffraction lines as due to quartz in the presence of other crystalline material than by the problems of measurement of the line.

It is evident that, with the very wide range of quartz contents observed, no satisfactory average proportion of the total respirable dust can be applied to many places in the mine. Though the quartz tends to be a more constant proportion of the ash, the quantity of ash at the threshold limit value[5] for 20% quartz or more is too small (less than 0.5 mg/m^3) to weigh with sufficient accuracy because of 'tare' changes during filter loading and unloading. The x-ray diffraction determination of quartz is much more sensitive and unaffected by 'tare' changes.

It should be noted that only crystalline forms of free silica can be detected by x-ray diffraction and of these only the most common, quartz, has so far been found and determined. The extension to the other crystalline forms is apparently easy and is waiting for the requirement.

Sampling Period

Very large variations in dust concentrations both within and between shifts were found; this suggests that the average of several shift period samples is required to estimate the miners' dust exposure and the efficiency of the dust control system. This position has been found in coal mining.[15]

Consideration of the biological and statistical sampling factors has suggested 10 days as an optimum sampling period for mineral dusts.[14] However, it is considered that, with present instrumentation and the requirement for collecting data for engineering control, a sampling period of one shift is a reasonable compromise.[2]

Personal Dust Sampling Versus Working Place Sampling

This work has shown that there are large and significant differences (up to 4:1) in the dust exposure of miners working nominally in the same place. This has been found in other types of mining as well.[15] On longwall coal mining there are a large number of men working in one place and it is worthwhile to study the variation between the miners and to select the most useful fixed position for sampling.[26] However, in hard rock mining there are usually only a few men in one working place and each man may have a large effect on his own dust exposure; thus there may be considerable advantages in changing to a routine dust sampling system based essentially on men instead of working places and involving the men in their own dust control far more than at present. These aspects need further study.

Mechanical Performance

The Casella Personal Gravimetric dust sampler, though not designed for use in mines, performed well except for the following problems: (a) harness was unsatisfactory so it was replaced by a miner's belt fitted with a shoulder strap; the sampler components were securely fastened to the harness; (b) the finger cotts in smoothers were prone to failure, apparently from exposure to sunlight; and (c) the pump case was damaged occasionally in narrow manways.

The most frequent maintenance required, at intervals of about 40 shifts, was replacement of pump valves and cleaning of the motor commutators.

The objectives of the dust sampling program in hard rock mines need careful reconsideration especially as large changes in the sampling method are being considered. Some useful reviews have been prepared for British coal mines,[15] German mines,[27] and U.S. coal mines.[28]

Conclusions

1. Using small, battery-powered size-selective samplers, gravimetric sampling and determination of quartz is feasible in hard rock mines.
2. Quartz determination on most samples is necessary in mines with medium- and high-quartz ores because of large variations in composition and greater sensitivity of the quartz assessment.
3. Because of the great variability in the dust concentration, shift period samples will provide a better esitmate of the dust exposure than will a few short period samples. Additional investigation is required to determine an adequate frequency for sampling.
4. It is feasible to issue personal gravimetric samplers to miners at the headframe, to operate them all shift without adjustment, and to collect them on return to surface.
5. The personal gravimetric sampler used in these tests suffered mechanical damage in some circumstances, so it is suggested that the combination of the sampler with the cap lamp battery[29,30] be tested underground.

Acknowledgements

The authors thank the McIntyre Research Foundation and the Mines Accident Prevention Association of Ontario for supporting this work, and Messrs. Chmara, Dix, Newkirk, Pullen, and Rutherford for the many helpful discussions held during the work and preparation of the reports.

The authors also thank the mines and the personnel involved without whose willing co-operation this study could not have been done.

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1. 5

Section 1—Chapter 5

A Combined Personal Sampler for Dust and Radon-Daughter Exposure in Mines

by

W.M. Gray, G. Knight and R.A. Washington

in Personal Dosimetry and Area Monitoring Suitable
for Radon and Daughter Products
Nuclear Energy Agency, Paris. pp127-134 (1976)

ÉCHANTILLONNEUR INDIVIDUEL COMBINÉ POUR ÉVALUER L'EXPOSITION À LA POUSSIÈRE ET AUX PRODUITS DE FILIATION DU RADON DANS LES MINES

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RÉSUMÉ

Un échantillonneur de poussière individuel destiné à être utilisé dans les mines de roche dure a été mis au point au laboratoire d'Elliot Lake. Il comporte les caractéristiques suivantes : classement des particules inhalables (alvéolaires), échantillonnage unique pendant un poste de travail complet, évaluation gravimétrique pour la poussière totale et analyse aux rayons X pour le quartz.

Une pastille thermoluminescente permettant de mesurer de façon cumulative le rayonnement alpha a été placée près du filtre dans l'échantillonneur de poussière. On peut ainsi évaluer l'exposition aux produits de filiation du radon.

Des méthodes d'échantillonnage possibles sont décrites brièvement.

A COMBINED PERSONAL SAMPLER FOR DUST AND RADON-DAUGHTER EXPOSURE IN MINES

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ABSTRACT

A personal dust sampler has been developed at the Elliot Lake Laboratory for use in hard rock mines. It features respirable (alveolar) dust size selection, single full-shift operation, gravimetric assessment for total dust and x-ray assessment for the quartz component.

A thermoluminescent chip for cumulative measurement of alpha radiation has been mounted adjacent to the filter in the dust sampler to provide for the assessment of exposure to radon daughters.

Possible sampling strategies are briefly discussed.

INTRODUCTION

This paper reports on progress in the development of a personal sampling instrument for the combined measurement of dust and radon-daughter exposure in mines. The new instrument is based on the Canadian Mining Personal Dust Sampler (CAMPEDS), which was developed at the Elliot Lake Laboratory for use in hard-rock mines.

The CAMPEDS was designed to allow the measurement of quartz* by x-ray diffraction as well as total dust. Experience in its use has shown that particulates from diesel exhaust and oil mists from compressed air operated equipment can also be measured successfully. Since radon daughters are akin to the lattter pollutants in being collectable on a filter it is logical to consider ways of measuring them on the same filter.

The CAMPEDS and its modification to allow the measurement of radon daughters will be described. Possible sampling strategies will be discussed briefly.

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* Quartz, a form of free silica, is a very common mineral and is particularly hazardous to health.

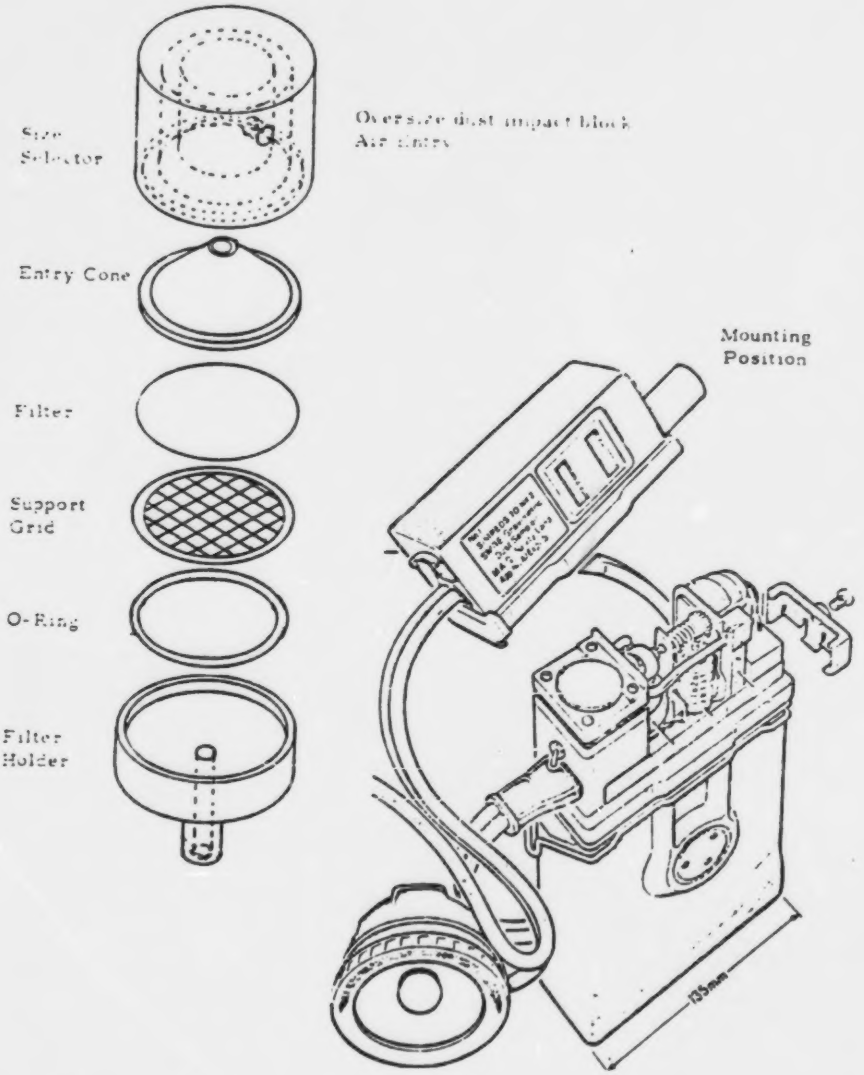
Size Selector
Oversize dust impact block
Air entry
Entry Cone
Mounting Position
Filter
Support Grid
O-Ring
Filter Holder
135mm

FIGURE 1: The Modified Filter Holder and Size Selector for the SIMPEDS Pump.

THE CANADIAN MINING PERSONAL DUST SAMPLER (CAMPEDS)

Between 1965 and 1969 the Elliot Lake Laboratory carried out a comparison of dust sampling instruments (1). It was found that there was a very wide variation in indications of relative hazard by different instruments in dust clouds of differing characteristics. Attention was then turned to the development of sampling and assessment techniques to give a better indication of the health hazard.

Preliminary studies (2, 3, 4) showed that full-shift portable self contained samplers could collect enough dust for assessment of total dust by weighing, and of quartz by x-ray diffraction. It was also found that a personal sampler indicated, on the average, a 25 per cent higher dust concentration than a fixed sampler at the nearest safe location in the working place. Thus the use of personal samplers would provide samples that were larger and therefore somewhat easier to assess and would simplify the logistical problem of taking full-shift samples for men scattered in small groups.

To fill the need for a suitable personal sampler, the CAMPEDS, sketched in Figure 1, was developed (4, 5). Its particular features are:

1. respirable size selection to simulate the deposition of dust in the alveolar region of the lungs where it offers the major health hazard,
2. full-shift period sampling,
3. combination with the cap-lamp battery to minimize the logistical problems of issuing it to underground miners,
4. a filter cassette to eliminate handling of the delicate filters outside the laboratory,
5. an impaction size selector, which is more compact and more readily machined reproducibly than other designs,
6. dust collection on a silver membrane filter for subsequent assessment by
    a) weighing (tare and gross) to give total respirable dust,
    b) ashing and reweighing to give respirable combustible dust or its complement, respirable mineral dust,
    c) x-ray diffraction to give respirable quartz dust.
    d) development of elemental (e.g. lead) analysis techniques is in progress.

The CAMPEDS has been used for investigations in a number of mines. Many of the problems associated with the use of such instruments in mines (6) have been overcome and CAMPEDS has been developed into a rugged instrument with satisfactory performance.

MEASUREMENT OF RADON DAUGHTERS BY MEANS OF THE CAMPEDS

Thermoluminescent dosimetry has been studied extensively by other investigators (7, 8, 9). It is based on the fact that certain materials, e.g., CaF_2 and LiF, are able to absorb energy from ionizing radiation and to release it again in the form of light when they are heated. The intensity of the light output is proportional to the cumulative total energy of the ionizing radiation. Thus, if radon daughters are collected on a filter to which a chip of such a thermoluminescent material is exposed, a measurement of the light output on heating of the chip can be made. After suitable calibration this measurement will yield the cumulative exposure to radon daughter in WLH* represented by the sample on the filter.

In the most recent studies of this process (10, 11) chips of thermoluminescent dosimeter (TLD) material (embedded in teflon) were exposed to airborne dust during its collection on a filter at a low flow rate (100 cc/min) for a 40-hour work week. Thus the total volume sampled was 240 l. In one of these studies (10) the following conclusions were reached:

"1. The air pump should be an integral part of a miner's lamp battery rather than a separate unit from the lamp battery. The air pump should be wired in such a manner that when a miner turns his lamp on the air pump is automatically turned on. Such an arrangement would obligate each miner to transport the monitoring system throughout his working area in a mine because a miner's light is usually the only source of illumination underground.

"2. The air pump must be completely sealed to prevent water and particulate matter from corroding and binding the moving parts of the pump.

"3. The filter-head unit should be designed as an integral part of the miner's lamp. The air inlet of the filter-head should be shielded from direct impaction of mud or other particulate matter.

"4. The entire unit should be as compact and light as possible.

"5. The final testing procedure should involve miner usage under actual mining conditions. Laboratory testing usually underestimates the severe and abusive treatment encountered by equipment in the mining operations."

It is intended to attempt to meet these requirements by incorporating a TLD chip in the entry cone of the CAMPEDS filter cassette (Figure 2). Because the CAMPEDS samples for an 8-hour shift at a flow rate of 2 l/min, however, the total volume sampled is 960 l. In order to keep the exposure of the TLD chip at approximately the same level as in the previous studies the entry cone has been modified to accommodate it in such a way that the chip is expected to "see" about one quarter of the dust sample on the filter.

The modified CAMPEDS, incorporating a TLD chip, is about to be calibrated and field tested. It is expected that a negligible fraction of the radon daughters will be associated with the coarse dust sizes rejected by the size selector, but this will be checked. Field tests will include the periodic measurement of WL by standard methods in parallel with the modified CAMPEDS, and correlation of the estimated WLH exposure with the integrated light output of the TLD chip indicated by the TLD reader.

One other possible technique for exposure extimation should be mentioned. During and after collection of a respirable dust sample, the associated radon daughters decay to Pb^{210}

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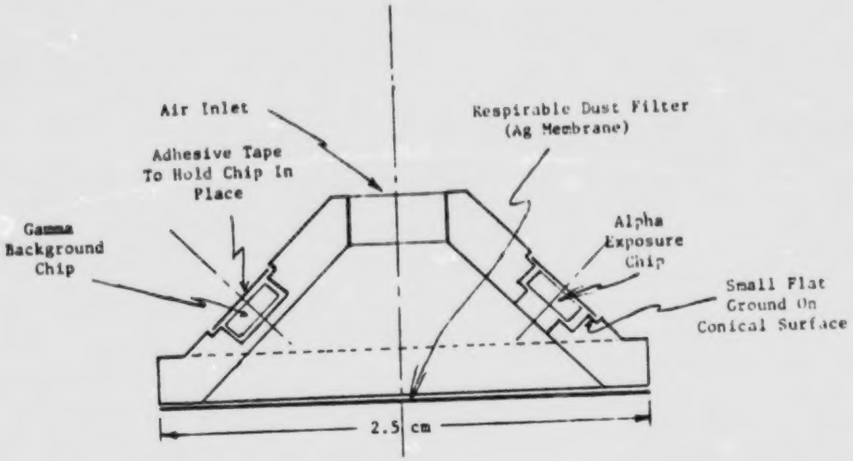
* WLH = Working Level-Hours, i.e. the product of the radon daughter concentration in working levels and the exposure time in hours.

FIGURE 2: Section Through Modified Campeds Entry Cone (Not to Scale)



(RaD, $t_{\frac{1}{2}}$ = 22 years). This in turn decays to Bi^{210} (RaE, $t_{\frac{1}{2}}$ = 5 days) and Po^{210} (RaF, $t_{\frac{1}{2}}$ = 138 days). The latter is an alpha emitter (E = 5.3 MeV) which may be measured as it grows into equilibrium with Pb^{210}. Preliminary tests on old (> 6 months) respirable dust samples have indicated that the technique may be practical, but much additional work needs to be done.

SAMPLING PROGRAMS

Routine sampling programs for dust and radon daughter measurement may have various objectives, such as engineering control, estimation of exposure for groups or estimation of exposure for individuals. The following three basic approaches have been suggested for meeting these objectives:

1. Short-period sampling, with periods of the order of minutes.

2. Full-shift sampling, either personal or by area.

3. Continuous monitoring, either personal or by area.

Short-period sampling, frequently repeated if necessary, is used for engineering control and the same type of sampling, repeated at intervals of the order of months, is the basis of the historical system for estimating personal exposure. The CAMPEDS is intended for exposure estimation using full-shift sampling. Holaday (6) has discussed the advantages of continuous personal dosimetry for radon daughters, but, unlike the film badge, the pump- and-filter samplers are expensive. Continuous monitoring is unlikely to give a sufficient improvement in exposure estimates over random full-shift sampling to justify the greater cost of the former.

Full-shift area sampling for dust is used in coal mines to monitor the exposure of the relatively large groups of men who work on long wall faces. This type of sampling cannot be equally satisfactory for uranium mines, where the men are scattered in small groups. In a practical monitoring system it may be necessary to calculate the individual's exposure from results obtained by

TABLE I

VARIABILITY OF SHIFT MEAN DUST CONCENTRATIONS FOR INDIVIDUAL MINERS

Occupation	No. of Shifts	Respirable Quartz Dust Concentration (normalized to mine average)*		
		Mean	Standard Deviation	Range
Shift Boss	6	0.8	0.4	0.5 - 1.7
Driller	5	1.1	0.4	0.6 - 1.5
"	6	1.6	0.3	1.0 - 2.7
"	3	2.5	2.2	0.3 - 5.3
"	3	1.4	1.6	0.1 - 3.7
"	4	0.6	0.1	0.4 - 0.8
Slusher	3	1.4	0.6	0.9 - 2.4
"	6	1.6	0.7	1.1 - 3.0
"	3	1.2	0.4	0.9 - 1.7
Muck & Drill	4	0.7	0.05	0.6 - 0.7
"	6	0.7	0.2	0.4 - 1.0
"	5	0.6	0.2	0.4 - 1.0
"	3	1.5	1.2	0.4 - 2.8
"	4	0.4	0.15	0.2 - 0.6
Pipefitter	6	1.7	0.7	0.6 - 2.5
"	6	1.2	0.5	0.6 - 1.7
Tracklayer	5	1.4	0.4	0.7 - 1.8
"	6	1.2	0.6	0.6 - 2.3
Haulage	5	0.6	0.2	0.1 - 0.8
"	4	0.7	0.2	0.5 - 1.0
"	5	0.5	0.15	0.3 - 0.7
"	4	0.6	0.05	0.5 - 0.6
Cage Tender	5	0.7	0.3	0.2 - 1.2
Mean Values		1.00	.5	-- --

* Expressed as a ratio to the average dust concentration found in this mine.

personal full-shift dosimetry on randomly selected shifts, combined with the results of similar measurements on other individuals doing similar work. Full-shift monitoring of the working places on randomly selected shifts may also be needed to minimize the amount of personal sampling required and to check the validity of the results.

It is well known that the details of a sampling program depend critically on the variability of the measurements in space and time, and on the accuracy with which the estimate of exposure is required. The latter factor must ideally be determined from epidemiological studies linking the susceptibility of workers, with its own inherent variability, to the particular type of exposure in question. The variability of the measurements must be established at the outset of the sampling program under the prevailing conditions of mine operation. All important parameters, such as those relating to ventilation, should be under control.

At the Elliot Lake Laboratory work is under way to obtain adequate data on the variability of dust and radiation exposure in the uranium mines. As part of this work the performance of the modified CAMPEDS will be throughly field tested. Through the design and testing of sampling programs to meet various objectives it is proposed to develop a basis for choosing practical programs for routine sampling.

Table 1 shows the high variability of full-shift quartz measurements observed for uranium miners sampled on 3 to 6 shifts doing nominally the same work in the same place.

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Discussion

It was suggested that the quantity of ^{210}Po on a dust sample would be too small to measure, the authors replied that it had been measured with a 16 hour counting period using a spectrometry in a preliminary test of the method and this view was supported by a further speaker

1. 6

Section 1—Chapter 6

Gravimetric Dust Sampling with Quartz Analysis and its Use in Metal and Mineral Mines

by

G. Knight and T.S. Cochrane

in International Mine Ventilation Congress,
Johannesburg, 1975
edited by R. Hemp and F.H. Lancaster pp407-414
Mine Ventilation Society of South Africa. Johannesburg

ÉCHANTILLONNAGE DE LA POUSSIÈRE AVEC DÉTERMINATION DE LA CONCENTRATION DE QUARTZ ET UTILISATION DANS LES MINES DE MÉTAUX ET DE MINÉRAUX

G. Knight et T.S. Cochrane

RÉSUMÉ

L'échantillonnage gravimétrique avec détermination de la concentration de quartz constitue le principe de fonctionnement d'un système de mesure de la poussière mis au point pour les mines de roche dure canadiennes. Le système comporte les caractéristiques suivantes :

a) classement des particules pour simuler la sélectivité des deux principales régions du poumon, les voies bronchiques et les alvéoles;
b) échantillonnage de longue durée pour obtenir une valeur de l'exposition pendant un poste de travail complet;
c) incinération directe sans changement de la tare, avec des filtres à membrane en argent de 25 mm de diamètre comportant des pores de 5 µm;
d) évaluation gravimétrique de la poussière totale recueillie;
e) détermination par diffraction des rayons X de la concentration de l'élément constituant le plus dangereux, le quartz.

Les résultats préliminaires montrent que l'échantillonneur de poussière individuel destiné aux mines canadiennes (CAMPLDS), mis au point pour être utilisé régulièrement dans les mines, présente certains avantages. Par ailleurs, les résultats montrent aussi qu'il est possible d'analyser aux rayons X des échantillons de poussière atmosphérique recueillis pendant un poste de travail complet et pesant moins de 0,5 mg.

Les problèmes associés au système de mesure ont été identifiés et les travaux de mise au point se poursuivent.

Gravimetric Dust Sampling with Quartz Analysis and its Use in Metal and Mineral Mines

By G. KNIGHT* and T. S. COCHRANE†

SYNOPSIS

Gravimetric sampling with quartz analysis is the basis of a dust measuring system developed for Canadian hard-rock mines. The system incorporates the following features:

(a) size selection to simulate the selectivity of the two major regions of the lung, the bronchial tract and the alveolae;

(b) long-period sampling to give full shift exposure;

(c) direct ashing without change in tare weight, using silver membrane filters of 25-mm diameter and 5 μm pore size;

(d) gravimetric assessment of the total dust collected;

(e) X-ray diffraction assessment of the most hazardous component, quartz.

Preliminary results indicate that the Canadian Mining Personal Dust Sampler (CAMPLDS) developed for routine use in mines has certain advantages. Also, the results show X-ray analysis of full-shift airborne dust samples weighing less than 0,5 mg is feasible.

Problem areas in the measuring system have been identified and development work is continuing

INTRODUCTION

At the Johannesburg Pneumoconiosis Conference[1] in 1959 world dust experts concerned with the measurement, control and effects of industrial dust recognized the need to improve dust sampling and assessment techniques. To obtain dust values suitable for correlation with clinical observations they recommended that a measuring system should have the following basic characteristics:

(a) aerodynamic size selection;

(b) the collection of only the respirable dust fraction;

(c) long-period sampling (full shift);

(d) gravimetric assessment of the dust collected;

(e) assessment of surface area of quartz dust in the sample.

Since that Conference, the measurement technology in the coal mines of Europe has advanced to the stage of development and acceptance of size-selective two-stage long-period dust samplers and gravimetric assessment of the respirable dust fraction.[2,3] However, no acceptable method for the assessment of the surface area of quartz particles has been developed.

In hard-rock mines similar advances in measurement technology have not been realized. It is difficult to develop dust sampling and assessment procedures that are applicable over the wide range, 0 to 100 per cent, of free silica (mainly quartz) and the related range of threshold limit values.[4] Successful application of a gravimetric procedure in hard-rock mines requires high accuracy in the handling and weighing of filters and a quick method of measuring the quartz content of the samples.

There are as yet no methods for measuring the surface area of quartz when mixed with other dusts; however, both infrared absorption and X-ray diffraction can be used to measure quartz in terms related to its mass or volume rather than surface area. The use of these techniques is encouraged by Goldstein and Webster's[5] conclusion, from intra-tracheal injection of sized quartz particles, that mass may be a better indicator than surface area of the harmful effect of quartz in the respirable size range.

PRELIMINARY STUDIES

Prior to 1970, thousands of dust measurements were taken in simulated dust clouds in the Mines Branch laboratory and in hard-rock mines using a variety of sampling instruments and assessment techniques. Mine environmental conditions were appraised and dust measurement systems evaluated.[6,7] General conclusions of primary significance for hard-rock mines were:

(i) The significant components of dust in the mine air are the total respirable dust, the respirable combustible dust, the respirable incombustible dust and the respirable quartz dust.

(ii) The correlation between number count and mass dust values is poor. Figure 1 shows a comparison between results obtained by the two techniques under laboratory and under mine conditions. It is found that the two techniques agree in classifying working places in high-quartz mines (65 per cent quartz in ore) as either acceptable or non-acceptable in 75 per cent of the cases, provided that 300 ppml is assumed to be equivalent to 0,1 mg/m^3 of respirable quartz. Any other conversion relation leads to agreement in less than 75 per cent of the cases.

(iii) The total respirable dust and the combustible and incombustible components can be determined by weighing and the quartz content by X-ray analysis.

(iv) Dust exposure can best be determined using personal samplers. Experiments were conducted where samplers of the same type were worn by a miner and set up at a fixed position in the working place. Results showed that the personal sampler often indicated dust concentrations as much as 50 per cent higher than that in a fixed position; the average indicated value was 25 per cent higher (Fig. 2). In short, there are considerable difficulties in using a fixed-position sampler to take samples that are representative of one man's exposure. While the relationship between each man's exposure and that of a

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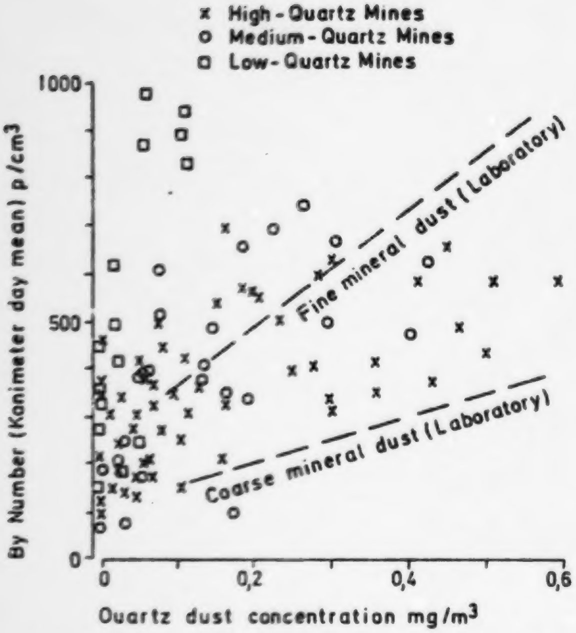

Fig. 1. Comparison of konimeter and respirable quartz dust concentrations.

fixed-position sampler could be determined, this would require a considerable amount of effort and is probably worthwhile only if there are a large number of miners to be monitored, for example, at a longwall coal face.[8]

(v) Gravimetric assessment is of little value for determining compliance with threshold limit dust concentration below 0,5 mg/m³ without developments in protection of filters during handling. The repeatability of weighing of unhandled filters is about 0,1 mg, of filters loaded and unloaded about 0,03 mg, and of those taken underground somewhat greater. Occasional errors of up to 0,2 mg occur due to loss of loose pieces of filter or to contamination. The weight of sample less than 0,5 mg needs to be verified by visual observation of the appearance of the dust deposit and scanned for absence of physical damage.

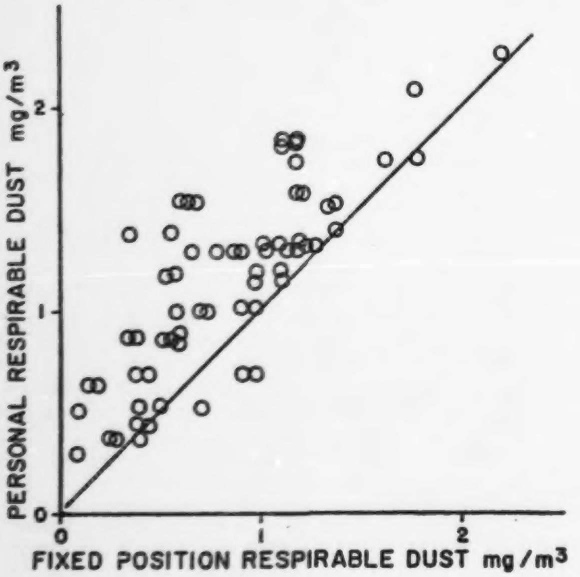

Fig. 2. Comparison of personal and fixed-position samples.

(vi) The percentage of quartz in respirable airborne dust varies greatly, mainly because the proportion of dust of mineral origin varies greatly. Thus the total respirable dust, even if it could be weighed accurately, would be a poor indicator of the possible hazard due to quartz.

DESIGN CRITERIA

As a result of experience to date the following criteria are proposed for an improved dust sampling system:

(a) Short-period sampling should be changed to full-shift personal dust sampling rather than to full-shift fixed-position dust sampling. This would have the advantage that the man's total job would be monitored.

(b) On the supposition that there would be a large number of samples, of the order of thousands per year, handling should be reduced to a minimum. Quartz analysis should be undertaken directly on the dust deposit collected on a filter.

(c) X-ray diffraction equipment has a wider range of capability than infrared absorption in analysis of all minerals and would in the final analysis provide a larger body of supporting data.[9]

CANADIAN MINING PERSONAL DUST SAMPLER (CAMPEDS)

The Casella personal gravimetric dust sampler[10] was the only instrument on the market which collected dust on 25-mm diameter filters and for this reason was chosen for preliminary experiments in collecting samples for mass and quartz determinations. Experience with the instrument showed that it was capable of collecting full-shift samples in most mining situations. The exception was in mines with high diesel usage where sampling often had to be limited to as little as three hours. This was not considered to be a serious long-term problem as the observation of high average dust concentrations of over 2 mg/m³ during a shift from diesel exhausts, led to research into their origin and recognition of the need for their control.[11]

The Casella sampler was mechanically adequate as a fixed-position sampler but not as a personal sampler for mine use. Four main problems were evident:

(i) the harness was not strong enough;
(ii) the pump was easily damaged;
(iii) the hoses pulled off readily; and
(iv) the instrument was too bulky.

The SIMPEDS dust sampler[12,13] which combined the pump with the cap lamp battery appeared not to show the disadvantages of the Casella. However, the concept of a filter protected by foam to collect the dust was not found to be applicable to direct X-ray analysis for quartz on the filter. Without the foam to collect any dust knocked off the filter, the chances of loss from the dust deposit were too high.

It was observed that underground the cap lamp battery gets much less rough usage than the cap lamp and consequently is a more suitable mounting for the sampling head. Also, a sampling head in the battery would result in less weight and drag on the safety hat. The question was 'Is a sample taken at cap battery level equivalent to or related to a sample taken at the breathing zone?' A few tests were carried out with miners wearing two dust samplers, one on the lapel and one on the belt.[8] Reasonable agreement was found between the two samplers provided neither sampler was covered by outer clothing. The cap lamp battery dust sampler was acceptable when worn outside all clothes and if mounted near the breathing zone on a bracket when riding.

It was decided to combine the best features of the Casella and the SIMPEDS and incorporate them into a cap lamp battery sampler. For identification purposes the sampler was called the Canadian Mining Personal Dust Sampler (CAMPEDS). Prototypes were built and tested in mines. The first CAMPEDS using a Casella 13040 cyclone was described in an earlier report.[7] A sketch of the second model using a smaller impactor size selector combined with a filter holder in a cassette and a pump design based on the commercial SIMPEDS is shown in Fig. 3. This second CAMPEDS has the advantage that the filter need only be handled in the laboratory, thus decreasing the chance of damage.

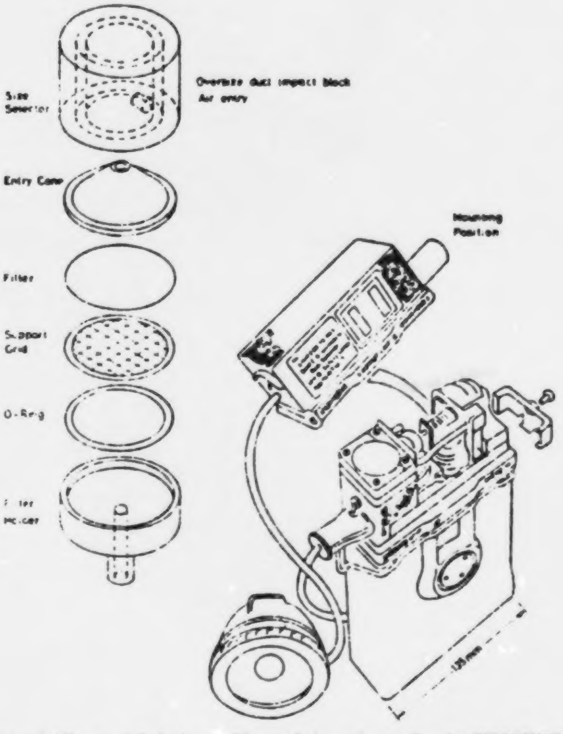

Fig. 3. The modified filter holder and size selector for the SIMPEDS pump.

The CAMPEDS as designed can be used for various purposes with very minor changes:

(a) normal design: 2 *l*/min with size selection equivalent to MRC specifications for most hardrock mining conditions. The CAMPEDS impact size selector has had its dimensions adjusted such that it gives the same estimate of respirable dust as does the Casella 113A gravimetric sampler with a horizontal elutriator[2] in a laboratory coal dust cloud with the respirable-sized fraction about 50 per cent of the total;

(b) low flow-rate design for high-diesel mines or other conditions where the filter overloads during a full shift; a smaller entry orifice and adjustment of the pump stroke; and

(c) total dust-collector design for assessment of materials such as arsenic in smelters; the same as (a) except that the impact block is omitted.

Further, the small sampling head casette could be fitted with any other pump with a suitable smoothed flow for fixed-position sampling in mines or elsewhere, or for personal sampling when cap lamps are not used. While the components of the CAMPEDS can be mass-produced cheaply in plastic, experience has shown that conductive materials are necessary to prevent electrostatic charges from altering the size selection characteristics of the sampler.

SAMPLE EVALUATION

Weighing techniques have been well developed for dust samples from coal mines. The silver membrane filters used in the CAMPEDS can be ashed to an accuracy of ± 0.03 mg by heating them on a stainless steel 'table' at a temperature of 480 to 500°C. The loss in weight, termed the 'respirable combustible dust', includes carbon dioxide lost from carbonates as well as organic materials. Weighing is done using a Mettler grammatic balance with a readability of 0,01 mg.

The starting point for studies on X-ray analysis was Dr. Leroux's work[14,15,16] on analysis of thin dust layers. Full details of the studies on X-ray analysis are given in a research report.[17] In brief, the studies consisted of the development of the X-ray technique in conjunction with the dust-sampling instrument modifications and sampling in mines.

Filter material and pore size

The choice of filter material and its pore size is a compromise between X-ray and dust sampling requirements. For X-ray analysis the requirements are low backgrounds under the quartz peaks, a linear calibration and a dust deposit of high surface density; for dust sampling, high collection efficiency, low air resistance, low moisture absorption, ease of handling, strength, and little or no weight loss on ashing. Two filter materials, namely, silver membrane and polycarbonate membrane, give low background levels under some of the stronger quartz diffraction lines; however, the polycarbonate is difficult to handle and the silver membrane is preferable on most counts. The large pore sizes, 3 and 5 *μm*, have the advantages of low resistance to airflow and of permitting the dust to be collected in a higher density deposit. However, while they are efficient in collecting all the dust, some dust at the start of sampling enters into the pores and is screened from the X-ray beam by the highly absorbent silver. This results in a non-linear calibration which is, however, more sensitive than the linear calibrations associated with the lower surface densities obtainable with the finer pore sizes, 0,8 and 0,4 *μm*.

Diameter and flow rate

The combination used is that which will permit the collection of the thickest deposits possible in a full shift without over-loading the pump and causing a significant drop in flow rate. It has been found that 25-mm diameter filters of 5 *μm* pore size can be used at 2 *l*/min in most metal mining situations for eight hours. A 22-mm diameter dust deposit is collected. When particulates from diesel engines exceed 2 mg/m^3, however, sampling times must be limited to three hours. Smaller-diameter filters can be used at this flow rate for a full shift on only a few sites and are thus not suitable for routine use.

X-ray wavelength

In selecting the X-ray wavelength, a compromise must be made between absorption, peak-to-background ratio, intensity and resolution. Absorption increases generally with wavelength and limits the thickness of sample for which a linear calibration is an acceptable approximation. The peak-to-background ratio for a given quantity of quartz determines the total count required for a given statistical accuracy and affects the sensitivity; generally, the peak-to-background ratio increases with increasing wavelength. The intensity of the X-ray output is inversely proportional to counting time for the same accuracy. High-output tubes are available with three target materials, Mo, Cu and Cr, which give X-ray wavelengths of about 0,7, 1,5 and 2,25 Å, respectively.

It is clear that a molybdenum target gives much less intensity and resolution than does a copper or a chromium target. The poor resolution using the molybdenum $K\alpha$ radiation increases greatly the problems of interference from other minerals; also, this radiation is applicable to a limited range of samples.

With Cu $K\alpha$ radiation, absorption exceeds only 25 per cent in full-shift iron-free samples if the mass exceeds 5 mg. With samples of mass up to this level, the underestimation of the quartz content is less than 25 per cent. In samples containing iron, 25 per cent absorption corresponds to an iron content of 0,5 mg.

Chromium radiation offers the advantages of greater resolution and greater peak-to-background ratio than copper radiation. However, it suffers from a slight loss in intensity and some decrease in its linear range due to increased absorption. Further studies are required to determine the extent of absorption problems and to develop a simple technique for absorption corrections following Leroux's method.[18]

X-ray settings

X-ray settings are a compromise between intensity and resolution. However, the gains in resolution that can be made by limiting the width of slits and the take-off angle are mostly minor compared with the great loss in intensity. The gain in resolution obtained by the use of 'Soller' slits in the incident beam is worthwhile at angles below 30 2θ but not at 45°2θ (2θ is the angle between the incident and the diffracted beams). If these slits were readily replaceable it would be advantageous to remove them at high angles. The diverging slit should be just large enough to illuminate the width of the dust deposit. The components of the tube tunnel have been machined to permit the use of the full 12-mm length of the electron beam on the target. The receiving slit is set to its maximum useful width, 0,4 mm, and a diffracted beam graphite monochromator is used to eliminate the $K\beta$ radiation and the possible high background caused by sample fluorescence. The monochromator gives a useful gain of 1,5 times in intensity over the use of an Ni filter to eliminate the $K\beta$ radiation. The step size used in step scanning is a compromise between the accuracy and the number of steps, with which is associated the time spent on computation. If the step size is too large the peak height varies with the relative position of the peak and the step. A step of 0,10°2θ with the wide receiving slit and the monochromator keeps this variation to within 10 per cent and is probably a maximum usable value. The value used, 0,08°2θ, was set by the equipment available, with simple modifications. A six-rise cam and clutched drive motor are used instead of the original components to minimize waste of time during stepping.

A scintillation detector was used with a wide window, 0,5 to 1,75 times peak value on the analyser. Narrow windows did not improve the peak-to-background ratios. Preset count times of 10 to 40 seconds are used and the data are printed on paper tape.

Calculation of quartz content

Calibration was carried out using samples collected in a pure quartz dust cloud to overcome the problems of uneven distribution and entry of dust into the pores of the filter and screening from the X-rays.[19] Permanent samples were prepared from a quartz-in-resin mixture and calibrated against the pure quartz samples. These permanent samples eliminate errors due to variations in X-ray intensity with time (the tube output decreases by 50 per cent during its useful life) and mechanical drift of the set-up.

Because the filter samples are occasionally distorted, it is not feasible to determine the peak at given angles and a computer program is used to:

1. find a statistically significant peak;
2. determine the shoulders;
3. select a background region on either side, rejecting adjacent peaks if any;
4. calculate net peak height, area and corresponding masses using the net peak height and area from the standard sample.

It was found that repeat measurements on one sample indicated about four times the error expected from the formula given by Klug and Alexander[20] and it appeared that one main factor causing discrepancy was statistical fluctuations in the shoulder positions.

FIELD RESULTS

General

A few hundred samples have been taken with the CAMPEDS prototype sampler which is combined with the cap lamp battery. It has been used in three underground mines with the mine staff issuing and receiving the samplers on surface and changing the filters. The results of a survey in one mine are shown in Fig. 4 as number of samples occurring in each concentration or per cent quartz range as marked. The distributions of dust concentrations are approximately log-normal and the characteristics are shown in Table I.

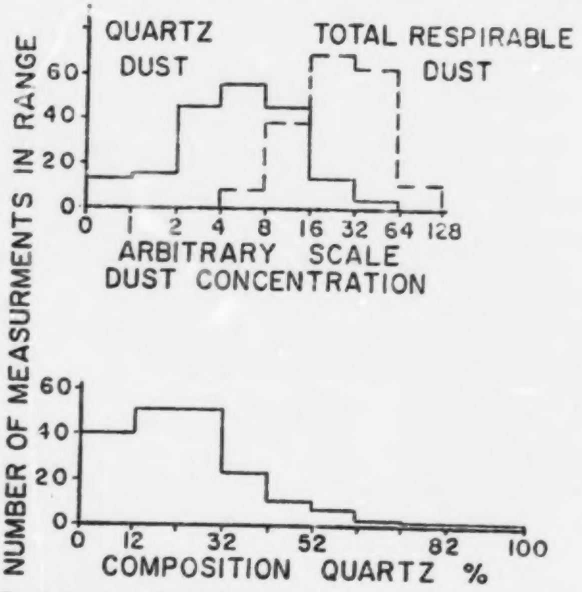

Fig. 4. Frequency distributions of respirable dust concentrations and composition found in one survey at one mine.

TABLE I

RESULTS OF A MINE SURVEY USING CAMPEDS

	Total respirable dust at ratio to mean	Respirable quartz dust as ratio to mean
Arithmetic mean	1	1
Median	0,85	0,74
20% exceed . .	2	2
10% exceed . .	2,3	3

Duplicate samples on personnel were not taken in this series and it is difficult to interpret the high results, that is, the 20 per cent exceeding twice the mean, and to decide whether they indicate random variations or that a few men were exposed to much higher dust concentrations than the average.

Composition of airborne dust in mines

Measurements were made of three components of the respirable portion of the dust, namely, combustible, ash and quartz. Figure 5 shows the results on samples from one mine. It can be seen that the results tend to lie on a line between 100 per cent combustible dust and 50 per cent quartz.

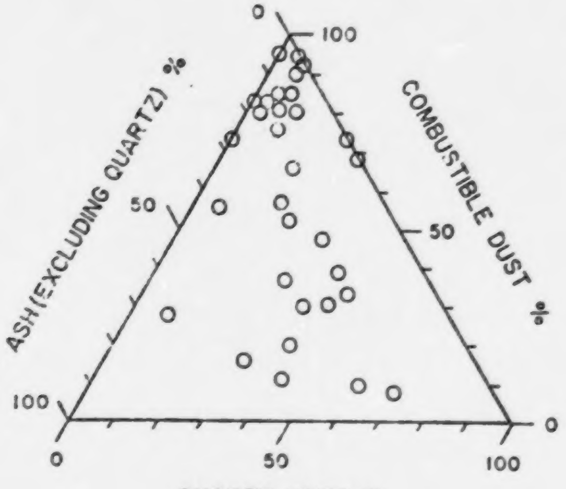

Fig. 5 Composition of the respirable portion of the airborne dust in a high-quartz mine.

The notable exceptions to this were samples in which the mass of ash was so small that weighing errors could account for the discrepancy. Similar results, with a different quartz percentage, were obtained in other mines. Table II shows the ranges and mean compositions found and compares them with the quartz in the ore.

TABLE II

COMPOSITION OF THE RESPIRABLE AIRBORNE DUST IN VARIOUS MINES: RANGES AND MEANS

Mine		Combustible Dust %	Ash %	Quartz as % of Total Dust	Quartz as % of Ash	Quartz in ore %
A	Range	—	—	0–100		99+
	Mean*	—	—	80		
	Mean*	—	—	50		
B	Range	—	—	0–100		65
	Mean	—	—	25		
C	Range	8–91	9–92	0–70	0–100	65
	Mean	57	43	22	52	
D	Range	5–100	0–95	0–15	0–30	30
	Mean	49	51	7,5	15	
E	Range	19–96	6–81	0–2	0–30	10
	Mean	50	50	1,5	3	
F	Range			0–15		15
	Mean			1,2		
G	Range			0–12		10
	Mean			1,7		

*The two means given here were obtained before and after the mean dust concentration dropped from 0,3 mg/m³ to 0,1 mg/m³ during a concentrated effort on dust control.

It is apparent that the measured compositions are very variable and that on the average the proportion of quartz in the total respirable dust is much less than that in the ore, and that in the ash is often less than in the ore. While errors in weighing account for some of the variation in composition, it is clear that the main factor is the lack of correlation between the different sources of dust. Table III shows results on the composition of dust produced in given operations, obtained by subtracting the concentration in the air entering the working place from those in the air leaving.

TABLE III

COMPOSITION OF THE RESPIRABLE DUST PRODUCED DURING SOME TYPICAL MINING OPERATIONS

Operation	Combustible dust %	Mineral dust %
Drilling (jack leg)	90	10
Mucking (comp. air)	80	20
LHD (diesel)	70	30
Slushing	10	90
Chute pulling	50	50
Crushing	n.d.*	100
Ore and waste passes	n.d.*	100

*n.d. – not detectable

Quartz analysis of airborne dust from mines

Interference has been found to be very common and is the factor controlling the sensitivity and accuracy of X-ray analysis.[18] To understand the X-ray results it is necessary to understand that there are two forms of error in calculated peak height or area due to interference by diffraction lines from other minerals. In form 1 the lines are close together, unresolved and result in a high estimate for quartz. In form 2 the lines are far enough apart to be resolved but the quartz line lies on the tail of the next and results in too high a value for the assumed linear background being subtracted, thus giving a low estimate for the quartz. Form-1 errors cannot usually be detected from the scan while the adjacent peaks giving form-2 errors are readily apparent if the interfering peak is sharp. However, if it is diffuse or multiple, it may result in non-obvious high background on one side of the quartz peak. While form-1 errors may be predictable from the minerals present, we have often found that after allowing for the known minerals in the sample there are one or more lines of medium intensity which have not been explained and could be due to unidentified components. On a few occasions one very strong quartz line has been found which is not confirmed on any other of the quartz lines. The most likely cause of this phenomenon is contamination by a few coarse quartz crystals of which one happens to be aligned to give a strong signal.

A selection of dust samples with the mass determined from four quartz diffraction lines (Miller indices 1, 0, 0; 1, 0, 1; 1, 1, 2; 2, 1, 1) is shown in Table IV. It is usually necessary to confirm an indication of a significant quantity by repeating the analysis on at least one other diffraction line.

DISCUSSION

It has been shown that long-period dust sampling with collection on a filter for subsequent gravimetric assessment and X-ray diffraction analysis for quartz is technically feasible in hard-rock mines. The gravimetric assessment is of value for non-mineral dust in all mines and for mineral dust in mines with low quartz content in the ore, but is insufficiently sensitive for mineral dust in mines with high quartz content in the ore.

TABLE IV

COMPARISON OF QUARTZ ANALYSES USING SOME OF THE DIFFRACTION LINES AVAILABLE

	Mass of Dust Estimated in mg using the Diffraction Lines Indicated							
	1, 0, 0		1, 0, 1		1, 1, 2		2, 1, 1	
Mine	Height	Area	Height	Area	Height	Area	Height	Area
A			0,017	0,019	0,025	0,024		
			0,030	0,031	—	—		
				0,014		0,015		0,001
				0,022		0,01		0,022
		0,01		0,01		0,06		0,01
B	0,023	0,049	0,006	0,007	0,007	0,002	0,010	0,003
	0,110	0,093	0,20	0,305	0,175	0,15	0,185	0,165
	0,185[1]	0,160[1]	0,064	0,077	0,057	0,053	0,050	0,041
C				0,24		0,00		
				0,32		0,27		
D		0,86		0,45		0,24[1]		0,60
		0,30		0,24		0,11[1]		0,26
E		1,03[2]		0,83[2]		0,36[1]	0,81	1,16
F		0,32		0,04		0,02		
				0,05		0,03		0,04
G				0,06		0,12		0,04
H		3,14		0,26		0,15		0,21
H′		0,24		0,21		0,16		0,14
H′ [3]		0,64		0,15		0,13		0,09

Footnotes:

[1] Adjacent peak detected.

[2] Percentage of quartz in the crushed ore determined by X-ray diffraction:

24% 64% 15% 35%

respectively, for each of the four lines. These figures compare with the 30 to 40 per cent found by other techniques.

[3] Sample H′ from a coal mine was ashed and re-analyzed with the results given in this line.

The change in instrument and period of sampling necessitate changes in the mine dust sampling procedures. These will be discussed under four headings: instrument performance, procedures, analysis of results (statistics) and dust standards.

Instrument performance

While the field trials of the CAMPEDS were judged to be successful, instrument problems did occur and may be identified as follows:

1. Acceptance. About 1 per cent of the men have refused to carry the dust sampler in this voluntary programme.
2. Contaminated samples. About 1 per cent of the samples appeared to have been significantly contaminated due to either inversion of the cyclone and transfer of the oversize dust to the filter or to deliberate dusting by personnel.
3. Mechanical failures. The most common has been a damaged lamp cord, the dust sampler portion has required little maintenance.
4. Underestimations. The dust concentration can be underestimated for two reasons:
   (a) The dust is knocked off the filter by rough handling. This has occurred on about 10 per cent of the very heavy samples and can readily be detected and allowances made.
   (b) The dust sampler is screened by clothing and air recirculates through the instrument. This can be overcome only by inspection and training.

Sampling procedures

The practice of using specialists to take dust samples and to record conditions has been generally successful. Short-period samples are taken and the samplers spend a short time at each working place. However, with long-period samplers, two visits to each working place are necessary to place and retrieve the instruments, and a third during the working period to record conditions. This means that the specialists are not able to cover as many working places.

Procedures for involving the miners or underground staff for placing and retrieving the samplers must be considered. During the course of our studies, miners were asked on occasion to place and retrieve samplers, usually with success, but sometimes samplers were forgotten and left underground. The placing and retrieval of dust samplers by the underground staff (shifters) in the Canadian mines does not fit in with their routine duties. The most reasonable way to obtain full-shift samples covering the working period would be to have personal dust sampling instruments worn by the miner. A few hundred samples have been taken in this fashion using either the separate sampler or the CAMPEDS prototype which is combined with the cap lamp battery. The procedures adopted by management staff for using the dust sampler should be as follows:

1. Issue sampler to miner at start of shift; check that it is running; note time.
2. Return of sampler by the miner to special lamp rack at end of shift; put on charge in normal manner.
3. Switch off samplers; note time; record miner's work, working place and time underground.
4. Record elapsed running time indicated by instrument.
5. Check and record flow rate through used filter.
6. Change filter cassette.
7. Check and record flow rate through clean filter.
8. If necessary, adjust flow rate or carry out other maintenance as required.
9. Designate miner to wear sampler for next shift.
10. Visit miner wearing the sampler during his working period both to check sampler and to note conditions for dust and ventilation control.

This dust sampling method with the CAMPEDS is a feasible routine dust sampling system for measuring the total personal dust exposure of a miner during one shift and for examining the whole job and not just the main operation.

Routine dust measurements as described would estimate dust exposure over the full shift. The requirements for dust sampling for engineering control still have to be considered. Having decided that a particular job required improved dust control, short-period samplers could be used to study its components. However, as the existing short-period samplers give very poor correlation with the gravimetric and quartz assessment techniques, it would be preferable to use the latter for dust control purposes. By running the long-period dust sampler continuously during one aspect of a particular job and turning off during others, it is possible to measure as little as 10 per cent of the required dust level, and thus break down the day's work into significant sections. Of course, to get meaningful measurements it is necessary to measure the dust in the intake air over the same periods unless it is very much lower than that produced by the operation.

Analysis of results

An accurate dust exposure estimate for one miner would require measurements over many shifts because of the great variability in dust concentration from one shift to the next, and would result in a tremendous mass of data for each mine. This is probably not necessary and the main requirement is to get reasonable estimates of exposure for each class of men, such as drillers, and for each section of the mine. The data may then be used for deciding on dust control requirements.

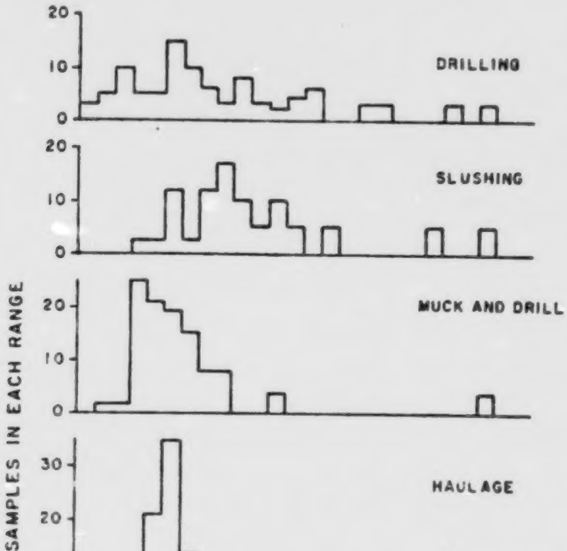

Fig. 6. Respirable quartz by occupations

It is clear that the results of dust surveys in a mine will indicate a very great variability in dust levels. It has been found that the highest shift-mean concentration is about 10 times the average shift-mean concentration. The distribution of concentrations approximate a log-normal distribution. The same variability is also apparent for most jobs in the mine when considered separately, as shown in Fig. 6. A study in which three to six personal samples were taken on each of about 20 men showed that the distribution of the means for individual men was about half as wide as the distribution for all the separate samples, that is, the maximum mean concentration for an individual man is about half of the maximum single-shift mean concentration (Table V, line 2). However, in this study, because of the small number of samples for each man, the highest mean concentrations were unduly influenced by the single high values included.

TABLE V

VARIABILITY OF SHIFT MEAN OF RESPIRABLE QUARTZ DUST CONCENTRATIONS FOR INDIVIDUAL MINERS WEARING PERSONAL SAMPLERS (A FEW SELECTED SAMPLES)

Number of shifts	Mean	Range
5	0,11	0,06 - 0,15
3	0,25	0,03 - 0,53
3	0,14	0,01 - 0,37
3	0,14	0,09 - 0,24
6	0,16	0,11 - 0,30
6	0,07	0,04 - 0,19
4	0,04	0,02 - 0,06
6	0,17	0,07 - 0,25
6	0,12	0,06 - 0,23
5	0,05	0,03 - 0,07

The following conclusions may be drawn from these surveys:

1. The average dust exposure of most men is less than twice the average shift mean dust concentration found.
2. Miners returning high dust concentrations, above twice the mean value, should be resampled to check that the exposure is not excessive.
3. The results should be averaged over each operation and and mine section to decide on the need for improved dust control.
4. The 10 per cent of shifts worked at high concentrations contribute some 25 per cent of the total dust exposure.
5. Attempts should be made to elucidate and control the factors leading to the high dust concentrations that occur on one shift in ten.

More samples are required to be able to undertake a complete statistical analysis of the variability of dust samples and to develop more than the *ad hoc* procedure given.

Dust standards

Dust standards have not been promulgated in Canada for the gravimetric dust sampling technique with quartz analysis. A review of existing standards throughout the world using both this and other types of dust sampling equipment suggests that upper and lower dust levels can be defined on which there would be general agreement. Concentrations above the upper level would be hazardous over a working lifetime and concentrations below the lower level would be safe. Target levels could be established for total respirable airborne dust in mines and for the two main constituents; for example

	Upper level	Lower level	Target
Total respirable dust	5 mg/m^3	1,5 mg/m^3	2 mg/m^3
Respirable combustible dust (diesel exhaust and oil mist not coal)	3 mg/m^3	0,5 mg/m^3	1 mg/m^3
Respirable quartz dust	0,5 mg/m^3	0,05 mg/m^3	0,1 mg/m^3

These target levels are those met with in the majority of mines visited and represent a dust level that can be obtained with present techniques. These dust concentrations assume a working lifetime of 35 years at eight hours per day, five days a week. For other working hours the concentrations should be adjusted to give the same exposure levels.

ACKNOWLEDGEMENTS

The authors wish to thank the personnel of the McIntyre Research Foundation, the Mines Accident Prevention Association of Ontario and the many mining companies, who were and are involved in these studies. Any advances made must be attributed to their co-operative efforts.

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CONTRIBUTION TO

GRAVIMETRIC DUST SAMPLING WITH QUARTZ ANALYSIS AND ITS USE IN METAL AND MINERAL MINES

(SESSION 13)

BY G. KNIGHT AND T.S. COCHRANE

It is encouraging to see that the thin film technique has reached such an advanced stage in Canada, as reported by Mr. Geoff Knight. Interference problems do not appear to have been worked out completely yet, but in any case a maximum value can be set to the estimate of quartz, that is, there might be less quartz but definitely not more than the estimate, apart from statistical variance.

The mass of the quartz would seem to be an acceptable parameter to measure since elutriation would tend to give a uniform size distribution in which case mass and surface area are completely correlated. The medical evidence seems to suggest that, for -7 μm quartz, mass is as important as surface area for predicting toxocity, but this is an aspect that deserves further investigation.

A.A. BRADLEY.

AAB/AMB

Section 1—Chapter 7

Mine Dust Sampling System–CAMPEDS

by

G. Knight

CANMET Report 78-7.
Energy, Mines and Resources Canada
Ottawa, Ontario

PREFACE

This is a record of experimental work at the Elliot Lake Laboratory in developing a method of dust sampling for mines using both gravimetric assessment and X-ray diffraction analysis for quartz. The field work undertaken in this connection is reviewed briefly.

The report emphasizes laboratory aspects of a dust sampling program using respirable dust samplers and X-ray diffraction analysis for quartz, and is intended mainly for staff setting up a sampling and XRD assessment system. It may also be useful to other personnel heavily committed to a dust sampling program. Report MRP/MRL 77-56, "Guide to gravimetric Sampling with Quartz Analysis in Mines" by the same author describes the field techniques of interest to most users of dust sampling equipment.

PREFACE

Ce rapport contient un compte rendu des recherches effectuées au Laboratoire d'Elliot Lake ayant pour but de mettre au point une méthode d'échantillonnage de la poussière dans les mines à l'aide de l'évaluation gravimétrique et de l'analyse par diffraction des rayons X (DRX) du quartz. On décrit brièvement les recherches à cet effet effectuées en chantier.

Le présent rapport met l'emphase sur l'analyse en laboratoire du programme d'échantillonnage de la poussière à l'aide d'un échantillonneur de poudre respirable et l'analyse du quartz par la diffraction des rayons X. Il a été rédigé à l'intention des personnes responsables de la mise sur pied de système d'échantillonnage et d'évaluation par la DRX et de celles qui s'occupent présentement d'un programme d'échantillonnage. Le rapport MRP/MRL 77-56 intitulé "Guide to Gravimetric Sampling with Quartz Analysis in Mines" du même auteur décrit les techniques employées en chantier et susceptibles d'intéresser ceux qui se servent d'un équipement d'échantillonnage de la poussière.

MINE DUST SAMPLING SYSTEM - CAMPEDS

by

G. Knight*

ABSTRACT

This report describes an improved method of sampling for air-borne dust in mines. Aerodynamic size selection is used to simulate dust deposition in the alveolar region of the lungs and dust is collected on a filter for periods of 2 to 10 hours. The dust is assessed by weighing before and after ashing to give estimates of total respirable dust and "respirable combustible dust". Respirable quartz dust is assessed on individual samples by X-ray diffraction. Silver membrane filters have been found most suitable for both gravimetric and X-ray diffraction analysis. They make it possible to measure directly without any transfer operation.

The X-ray diffraction analysis of quartz is improved by using the most intense beam possible with wide slits. The graphite diffracted-beam monochromator increases intensity and peak-to-background ratio. The choice of X-ray target material is a compromise between intensity, resolution and absorption with cobalt being the most suitable. More than one X-ray diffraction line is used to minimize the effects of interference.

Because of uneven dust deposition over the filter and uneven illumination by X-rays, the X-ray diffraction must be calibrated against the mass of quartz on samples collected with the sampler in a pure quartz dust cloud. Permanent quartz standards are required to eliminate effects of secular variations in X-ray intensity.

A robust personal dust sampler combined with the cap lamp battery called CAMPEDS was developed for convenient personal sampling in mines. The respirable size-selection characteristics of dust samplers are affected by small changes in dimensions and layout of components. It is essential that calibrations on complete instruments be carried out to test both the design and reproducibility of manufacture.

Key words: Dust Sampling; Full Shift; Personal Sampling; Quartz X-ray Diffraction.

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METHODE D'ECHANTILLONNAGE DE LA POUSSIERE DANS UNE MINE – CAMPEDS

par

G. Knight*

RESUME

Le présent rapport décrit une méthode améliorée d'échantillonnage de la poussière dans l'air d'une mine. La méthode de sélection aérodynamique selon la granulométrie est employée afin de simuler le dépôt de la poussière dans la région alvéolaire des poumons. La poussière est accumulée sur un filtre pendant 2 à 10 heures. Cette poussière est pesée avant et après avoir être réduite en cendre pour estimer la quantité totale de poussière respirable et de "poussière combustible respirable". La quantité de poussière de quartz respirable est mesurée sur chacun des échantillons par la diffraction des rayons X. Les filtres composés d'une membrane d'argent conviennent mieux aux analyses gravimétriques et de diffraction des rayons X. Par ce fait il est possible de mesurer directement sans effectuer de transfert.

On améliore l'analyse du quartz par la diffraction des rayons X en employant un faisceau lumineux le plus intense possible et des fentes plus larges. La méthode avec monochromateur graphite à faisceau diffracté fait augmenter l'intensité et le rapport sommet à fond. Le choix du matériau cible pour l'analyse aux rayons X nécessite un compromis entre l'intensité, la résolution et l'absorption. Le cobalt est la meilleure cible. On utilise plus d'une ligne de diffraction des rayons X afin de minimiser les effets d'interférence.

Comme la poussière ne se dépose pas uniformément sur le filtre et que l'illumination par les rayons X est inégale, la diffraction des rayons X doit être étalonnée selon la masse du quartz sur les échantillons prélevés d'un échantillonneur placé dans un nuage de poussière de quartz pur. Les normes de quartz permanentes sont requises afin d'éliminer les effets des variations séculaires de l'intensité des rayons X.

On a donc mis au point un échantillonneur robuste et personnel appelé CAMPEDS combiné à la pile de l'ampoule du casque. Les caractéristiques de sélection granulométrique des particules respirables des échantillonneurs de poussière sont affectées par les menus changements de la dimension et de la disposition des composantes. Il est essentiel que les réglages sur les instruments soient effectués afin de vérifier la conception et la facilité de reproduction.

Mots-clés: Echantillonnage de la poudre; échantillonnage personnel; diffraction des rayons X du quartz.

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CONTENTS

		Page
PREFACE		i
PREFACE		i
ABSTRACT		iii
RESUME		iv
CHAPTER 1 - INTRODUCTION		1
1.1	Deposition of Dust in Lungs	1
1.2	Clearance of Dust from Alveolar Region	2
1.3	Biological Effect of Dust in Lungs	2
1.4	Requirements for Dust Sampling System in Hard Rock Mines	3
1.5	Methods of Analysis for Quartz and/or Free Silica	4
	1.5.1 Wet chemical	4
	1.5.2 Infrared absorption	4
	1.5.3 X-ray diffraction	4
	1.5.4 Differential thermal analysis	4
	1.5.5 Choice of quartz analysis technique	4
1.6	References	5
CHAPTER 2 - OUTLINE OF DEVELOPMENT		7
CHAPTER 3 - Quartz Analysis		9
3.1	Permanent Quartz Standards	9
3.2	Calibration	10
3.3	Interference	11
3.4	Sample Absorption Studies	14
3.5	Summary of Quartz Analysis	19
3.6	References	20
CHAPTER 4 - FILTER CHARACTERISTICS		23
4.1	Dust Collection Efficiency	23
4.2	Site of Dust Deposition	23
4.3	Airflow and Pressure Drop	24
4.4	Shape and Size of Dust Deposit	24
4.5	Background for X-ray Diffraction Analysis	25
4.6	Hygroscopicity of Filter Material	25
4.7	Ashing	26
4.8	Choice of Filter	27
4.9	References	28

CHAPTER 5 - DUST SAMPLERS 29

5.1 Respirable Size Selection 29

5.2 Sampling Position 30

5.3 Filter Holders 31

5.4 Flow Smoothers and Flow Measurement 31

5.5 Sampling Pumps 32

5.6 References 33

CHAPTER 6 - DISCUSSION 35

6.1 Dust Sampling 35

6.2 Gravimetric Assessment of Dust 35

6.3 X-ray Analysis 36

6.4 Sampling and Analysis Time Study 37

6.5 Composition of Airborne Dust 38

6.6 Further Developments 40

6.7 References 41

ACKNOWLEDGEMENTS 42

APPENDIX A - DEVELOPMENT OF X-RAY INSTRUMENTAL TECHNIQUES.. 43

A.1 Filter Mounting 43

A.2 Generator and Ancillary Equipment 43

A.3 X-ray Tube 43

A.3.1 Intensity of characteristic radiation 44

A.3.2 Peak-to-background ratio 45

A.3.3 Resolution 45

A.3.4 Absorption 46

A.4 Take-off Angle 46

A.5 Beam Height 47

A.6 Vertical Divergence of Incident Beam 47

A.7 Diverging Slit 47

A.8 Receiving Slit 49

A.9 Step Size 49

A.10 Diffracted Beam Monochromator 50

A.11 Detector 50

A.12 Evenness of X-ray Illumination Over Sample 51

A.13 Noise Suppression 53

A.14 Comparison of Angle and Energy Resolution Systems 53

A.14.1 Experimental technique 53

A.14.2 Results 54

A.15 Data Printout 55

A.16 References 57

APPENDIX B - CALIBRATION OF QUARTZ ANALYSIS 59
  B.1 Preparation of Pure Quartz Samples in Laboratory Dust Chamber 59
  B.2 Calibrations of Quartz Samples 59
  B.3 Calibration of Other Dust Sampling Instruments Used in Elliot Lake Laboratory 61
  B.4 Effect of Quartz Dust Size 63
  B.5 References 64

APPENDIX C - COMPUTER PROCESSING OF DATA 65
  C.1 References 65

APPENDIX D - PREPARATION OF ROCK SAMPLES FOR ANALYSIS 71

APPENDIX E - Varieties of Free Silica 73
  E.1 Conclusions 75
  E.2 References 75

APPENDIX F - CALIBRATION OF CHARACTERISTICS OF SIZE SELECTIVE DUST SAMPLERS 77
  F.1 Dust Collection Specifications 79
  F.2 Test Methods 80
  F.3 References 82

APPENDIX G - DEVELOPMENT OF THE CANADIAN MINING PERSONAL DUST SAMPLER (CAMPEDS) 85
  G.1 Impaction Size Selector 85
  G.2 Laboratory Tests on CAMPEDS Sampler 87
  G.3 Underground Tests 90
  G.4 Pulsation Dampers 93
    G.4.1 Effect of pulsation dampers on dust collection 94
    G.4.2 Effect of pulsation on reading of taper tube flowmeter 96
    G.4.3 Discussion on pulsation dampers 96
  G.5 References 99

APPENDIX H - ANALYSIS OF ERRORS IN GRAVIMETRIC AND X-RAY DIFFRACTION ASSESSMENTS 101
  H.1 Weighing Errors 101
  H.2 Errors in Gravimetric Assessment 101
  H.3 Errors in X-ray Assessment 105

FIGURES

1.1 Dust deposition in lungs showing range of variation between individuals and different breathing rates. After Hatch and Gross (2) 2
3.1 Schematic of effects of absorption and penetration of dust into filter at start of sampling 11
3.2 Interference effects of adjacent diffraction line on line area estimation 12
3.3 Absorption of CuKα X-rays in dust layer as measured by intensity of silver 1,1,1 peak 16
3.4 Effect of absorption on intensity of quartz peaks in 3.2 pyrite-quartz dust deposit 17
3.5 Intensity of silver peak measured through dust deposit on field samples - copper radiation 17
3.6 Intensity of silver peak measured through dust deposit on field samples - chromium radiation 18
4.1 X-ray diffraction patterns from clean filters ... 26

A.1 Filter holder for automatic sample changer 44
A.2 Effect of Soller slits on diffraction lines from quartz 48
A.3 Pulse size distribution from X-ray scintillation detector 51
A.4 Contours of diffracted beam intensity from a 3-mm diameter quartz sample - 1,0,1 line CrKα radiation 52
A.5 Comparison of energy and angle resolution for samples with high and low densities of quartz - 1,0,0 diffraction line of quartz 55
A.6 Comparison of energy and angle resolution of samples of high and low densities of quartz - 1,0,1 diffraction line of quartz 55
A.7 Comparison of energy and angle resolution of samples with high densities of quartz - 1,1,2 diffraction line of quartz 56
A.8 Energy resolution chart for region covering the 1,1,2, 2,0,2 and 2,1,0 diffraction lines of quartz for samples with high and low quartz 56
B.1 Quartz calibration of X-ray with Casella samples 60
B.2 Quartz calibration of X-ray with Casella samples 61
F.1 Accuracy of rotameter reading on pump flow test.. 81
F.2 Leakage tests 81
F.3 Flow and pressure-drop test 82

G.1 The pump set up for adjusting flow rate 86
G.2 Filter holder and size selector for the CAMPEDS.. 86
G.3 Size distribution of particles determined by Coulter Counter 88
G.4 Efficiency of collection of particles by size selectors determined by Coulter Counter 89
G.5 Effect of mine depth on pump characteristics 90
G.6 Pulsation damper for Casella Model B personal dust sampler 97
G.7 Pulsation damper and cyclone-filter holder for mounting on cap lamp battery 98
H.1 Distribution of errors associated with weighing.. 102
H.2 Distribution of negative errors in gravimetric assessment calculated from samples showing high quartz contents 103
H.3 Gravimetric assessment error plotted against calculated mineral weight 104
H.4 Cumulative probability curves of weighing and gravimetric assessment errors 105
H.5 Differences between duplicate measurements of quartz 107

TABLES

2.1 Dust collection characteristics of samplers 7
3.1 Variations in X-ray intensity with time 10
3.2 Thin sample correction factors 11
3.3 Ratio of intensities of quartz diffraction lines 13
3.4 Comparison of quartz analyses using some of the diffraction lines available 14
3.5 Calculated mass limits for 20% departure from linear calibration with a selection of materials and radiations 16
4.1 Dust passing through filter materials 24
4.2 Dependence of body deposition of dust on filter pore size 24
4.3 Characteristics of filter papers relevant to quartz analysis for the six strongest quartz lines 27
5.1 Size selector comparisons 30
5.2 Comparison of lapel and belt sampling positions.. 31
6.1 Time study of analysis procedures 38
6.2 Composition of the respirable airborne dust in various mines: ranges and means 39

A.1 Intensities of diffraction lines (peak heights) relative to those using a copper target by net peak height 45

A.2 Net peak height to background ratio relative to that with copper target 45

A.3 Width of diffraction lines 46

A.4 Absorption in sample - intensity of silver lines relative to those given by the absorbed radiation 47

A.5 Effect of vertical divergence 48

A.6 Effect of receiving slit width 49

A.7 Effect of step size and position relative to the peak maximum 50

A.8 Comparison of graphite monochromator and nickel filter using CuKα radiation 50

A.9 Results from pure quartz sample.................. 54

B.1 Calibrations of quartz samples (1,0,1 line) for copper and molybdenum Kα radiation 59

B.2 Calibration of permanent quartz standards 62

B.3 Calibration of dust sampling instruments 63

B.4 Effect of particle size on diffracted intensity of X-rays from quartz 64

D.1 Calibration of pure mineral standards: usable diffraction lines 72

E.1 Description of free silica samples 73

E.2 X-ray intensities of silica varieties 74

F.1 Performance of size selector - dust sampler 78

G.1 Effect of diameter and number of orifices on the characteristics of an impact size selector 88

G.2 Tests on dust sampler No. 1 91

G.3 Tests on dust sampler No. 2 91

G.4 Tests on dust sampler No. 3 92

G.5 Tests on dust sampler No. 4 92

G.6 Tests on dust sampler No. 5 93

G.7 Effect of pulsation dampers on dust collection with the Casella personal gravimetric dust sampler 95

G.8 Effect of pulsation dampers on flowmeter reading Casella personal sampler pump 95

H.1 Distribution of random errors in weighing 102

H.2 Statistical linear regression analysis of X-ray intensity (peak area) from one quartz standard .. 106

CHAPTER 1

INTRODUCTION

Airborne dust that can be inhaled and deposited in the lungs of miners represents a potential health hazard. Measurements are required to indicate the extent of this hazard and the need for dust control. Such measurements should include the determination of physical and chemical parameters of the dust cloud and should relate to its biological effects.

The development of suitable measuring methods forms part of the Minerals Research Program of the Department of Energy, Mines and Resources. It developed from previous studies in which a number of, types of dust sampling instruments were compared when used in various laboratory dust clouds (1). It was found that for one type of cloud at various concentrations, any two types of instrument would indicate the same ratio of concentration within reasonable limits of ± 10%, although for different clouds — varying in type of material and particle size distribution — the indicated ratio of concentration ranged at least from 1:1 to 4:1. Thus one instrument could assess one working place as, say, twice as hazardous as another, whereas a second type of instrument could reverse the relationship. Clearly, all the instruments cannot give an adequate single-parameter estimate of the health hazard; the choice of a suitable instrument requires consideration of biological effects.

Dust sampling should be directed toward estimating the potential hazard of pneumoconiosis in its many forms — silicosis, anthracosis, etc. To do this, it is necessary to consider the physiological processes involved and to measure dust in terms of appropriate parameters. Factors to be considered in relating the onset of pneumoconiosis to the characteristics of the dust cloud are:

1. deposition of dust in the lungs,
2. clearance of dust from the lungs,
3. biological effect of dust in the lungs.

1.1. Deposition of Dust in Lungs

The only dust that can be deposited in the alveolar region of the lung where it may lead to pneumoconiosis, is that which remains in the inhaled air after passing through the respiratory tract or mucus-swept airways. Three physical mechanisms — impaction, gravitational settling and diffusion — are instrumental in depositing dust in both the respiratory tract and the alveolar region, and one appropriate parameter for describing the behaviour of a dust particle is its "aerodynamic size". This is defined as the diameter of a sphere of unit density having the same settling velocity as the particle in question.

The general dependence of alveolar and respiratory-tract deposition on size is shown in Fig. 1.1; however, precise values vary with the breathing pattern and with the individual (2). The extent of the variability is indicated by the hatched areas. In general, a minimum deposition occurs for particles about 0.5 µm in size. Impaction and gravitational settling increase with particle size and are responsible for most of the deposition at sizes above the minimum. Diffusion deposition increases inversely with size and is the main mechanism below 0.5 µm size.

To define the alveolar-deposition potential of an airborne dust cloud, size distribution should be specified in terms of aerodynamic size. Other measures of particle size may give misleading results, e.g., coal particles with an aerodynamic size of 5 µm may have projected areas equivalent to that of 5- to 15-µm diameter circles (3).

Size selectors have not been designed that simulate the entire effect, i.e., the increased deposition of very small particles, the minimum at 0.5 µm, the maximum at 1 µm, and the decrease at larger sizes. However, in airborne dust clouds resulting from mineral breakage, most of the mass and surface area of the dust is associated with the coarser portion — i.e., that part of the respirable dust above 1 µm — and most size selectors have been designed to simulate the alveolar dust deposition in the size range above

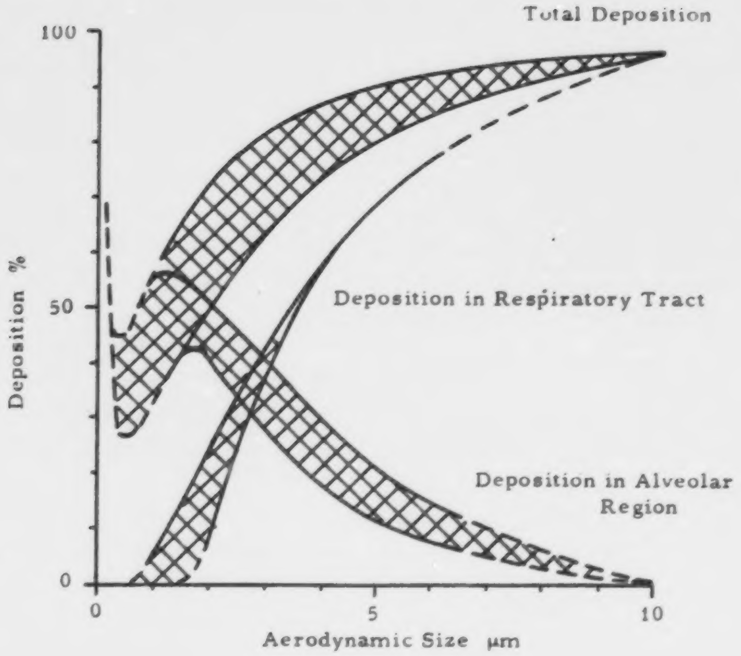

Fig. 1.1 - Dust deposition in lungs showing range of variation between individuals and different breathing rates. After Hatch and Gross (2).

1 µm, with a maximum efficiency of nearly 100% rather than the 40% shown in the figure. Usually all particles finer than this are collected with high efficiency and are included in the "respirable" dust portion.

Aerodynamic size selection should be used on all dust samplers for estimating the pneumoconiosis hazard, because of the difficulty of ascertaining the original state of aggregation and density after collection (4).

1.2. Clearance of Dust from Alveolar Region

The mechanisms of clearance are not as clearly understood as those of deposition. Experimental studies, reviewed by Hatch and Gross, have suggested that clearance depends on composition, size distribution, shape, and concentration of the deposited dust (2).

Experimental studies (2) have suggested that the clearance has a half-life of 20 days or more, and only at extremely high dust concentrations has there been suggestion of a breakdown. This slow rate of clearance suggests that the average dust exposure over a period of at least 20 days would be more significant than peak exposures, unless these are extremely high; however, the most relevant parameters for expressing these factors are not known. As a first approximation, dust exposure should be estimated as an average over long periods, not as peak concentrations.

1.3. Biological Effect of Dust in Lungs

The physical and chemical factors determining biological effects are: concentration, size distribution, composition, shape and residence time. The composition of dust is very important as shown by the very great differences in recommended threshhold limit values (TLV), such as 2 mg/m^3 for coal mine dust and 0.1 mg/m^3 for pure quartz dust (5). Other minerals which have low TLV's are other crystalline and amorphous varieties of silicon dioxide, asbestos, talc, and other fibrous minerals. In mining, quartz is the most common and wide-spread of these more harmful substances, both in the pure form and mixed as fine crystals with other minerals.

This report is concerned with methods of analyzing dust samples for quartz and with methods of collecting suitable samples of airborne dust in the respirable size range. Clearly, the choice of physical parameter — number, surface area, or mass — to be assessed has a great effect on the relative assessment of dust concentration in atmospheres of differing dust size distributions, and the parameter chosen should be related to the biological effect.

It has been shown that the health hazard of coal mine dust is closely related to mass in the respirable size range (6). It is widely assumed that mass is also the best measure for other dusts which do not give rise to a specific biological reaction. For free silica, the position is not clear and it is assumed on the basis of both theories of the physiological action of silica, that the surface area of the silica dust in the respirable size range is most closely related to its health hazard (4). However, some experiments on the intratrachael injection of sized quartz particles suggested that mass may be a better indicator of the silicogenic effect than is surface area (7).

At present no method exists for specifically measuring surface area of the silica in airborne dust and all the available analytical techniques measure parameters that are more closely associated with the mass than with the surface area of the silica particles. Page-Shipp and Harris concluded there is a high correlation between respirable surface area and respirable mass of airborne dust in South African gold mines as determined by microscopy on ashed thermal precipitator slides (8).

In routine dust analysis it is not practicable to measure a large number of parameters, and it is suggested that free silica, (quartz) should be specified by the single parameter, mass, as determined by an analytical method. However, because of doubt as to which parameter is most closely related to the biological activity and the effect of its admixture with other components, detailed analyses of size distribution and composition should be made in research studies on a range of samples of airborne dust. A suitable start might be to collect routine gravimetric respirable dust samples, bulking those from each job in each mine or section and analyzing them to obtain at least the following additional information: size distribution of all respirable dust, size distribution of respirable quartz dust, major mineral components, trace elements, and major organic constituents, as well as the form in which free silica occurs.

1.4. Requirements for Dust Sampling System in Hard Rock Mines

Consideration of the physiological processes described above led to the following requirements on which a new dust sampling technique should be developed for routine use in hard rock mining:

1. aerodynamic size selection to match deposition of dust in the alveolar region of the lung,
2. long-period sampling — a full shift is a convenient time interval,
3. quartz analysis to be carried out on individual dust samples.

Further non-physiological requirements are:

4. sampling instrument should be readily portable and self-contained,
5. sensitivity of dust assessment should be about one tenth of the appropriate threshhold limit values,
6. repeatability of the method need be only 10 to 20% because of the great variability of the airborne dust clouds in mines.

Dust sampling instruments meeting requirements 1, 2 and 4 have been developed for coal mines and iron foundries respectively (9, 10). These were examined as to suitability for collecting samples for quartz analysis and some were purchased. Requirement 3 is considered in detail in the next section.

1.5. Methods of Analysis for Quartz and/or Free Silica

Free silica or silicon dioxide crystallizes in a number of forms — α-quartz, high quartz; low, middle, and high tridymite; low and high crystobalite; keatite; coesite; stiskovite, and also occurs in non-crystalline form as glasses (11). These forms affect the analytical techniques to various extents. While all are more hazardous than combined silica (silicates) in most of its forms, α-quartz is the most common and its occurrence can be separated into two classes: coarse-crystalline (amethyst, etc.) and fine-crystalline (chalcedony, agate, flint, chert, jasper, etc.). The latter may have to be distinguished when certain of the analytical techniques are used.

A number of analytical techniques have been proposed and some have been extensively used:

1.5.1. Wet chemical

This technique separates free silicas from silicates by their relative solubilities in hot phosphoric acid. Almost all silicates are dissolved more quickly than the free silicas. The original technique used gravimetric assessment of the undissolved silica but more recent developments have used colorimetric methods of assessing the silica after dissolving the separated silica in hydrofluoric acid and have achieved sensitivities down to 10 μg of free silica (12). In assessing this method, it must be remembered that some silica dissolves in the hot phosphoric acid from the surface of each free silica particle, and that the complete solution of the large particles of silicate is not achieved. The time and temperature of phosphoric acid attack is set to balance these two errors; however, the balance achieved is dependent on the particle size distribution and on the varying solubilities of the silica and silicate varieties present. The technique requires very careful control of time and reagent purity to achieve consistent results (12), and has been used extensively in some laboratories.

This method is believed applicable to all varieties of free silica.

1.5.2. Infrared absorption

Certain infrared absorption spectral lines are specific to various chemical bonds, and the doublet at 12.5 μm wavelength has been widely used for silicon dioxide analysis. The usual technique has been that of mixing the finely-ground dust with a transparent salt (KBr), pressing the mixture into a pellet, and measuring the absorption. Recent work has shown that absorption can be measured directly on a dust deposit as collected on a filter, with a sensitivity of about 10 μg (13).

Some minerals interfere with the silica assessment and ashing is recommended for coal dust to reduce interference (13, 14).

The method is believed to be applicable to all forms of free silica.

1.5.3. X-ray diffraction

X-ray diffraction can be used for qualitative and quantitative analysis of most crystalline materials. The strongest diffraction line, that from the 3.34 Å spacing in the quartz crystal, has been widely used for quantitative analysis. While many materials interfere with this, qualitative analysis in conjunction with the extensive tables available has normally been used to identify possible interferents (15, 16). The technique has been developed to a sensitivity of less than 10 μg, and the method is applicable to all crystalline varieties of silica.

1.5.4. Differential thermal analysis

Quartz and the other crystalline varieties of free silica undergo marked phase changes with emission or absorption of heat at specific temperatures. This effect can be used to analyze them quantitatively; so far the sensitivity has been considerably less than the best achieved by the other methods listed above (17).

1.5.5. Choice of quartz analysis technique

It was expected that, if a method were

developed for use in mines, there would be thousands of quartz analyses to be done each year and therefore techniques using as little handling as possible should be tried before examining transfer techniques which might reduce the compromises required between dust sampling and assessment, and possibly give greater sensitivity and accuracy.

Preliminary studies on quartz collected directly on silver and vinyl membrane filters using both X-ray diffraction and infrared absorption were undertaken at other laboratories within CANMET. It was found that both techniques had sensitivities of about 0.1 mg quartz on a 25-mm diameter filter. The Elliot Lake Laboratory elected to purchase X-ray diffraction equipment because of its applicability to qualitative and quantitative analysis of other minerals as well as of quartz. The starting point for the X-ray diffraction technique was J. Leroux's excellent studies in Ottawa (18 to 22), based on the thin-film technique which largely avoids the need for corrections due to absorption in the matrix. Dr. Leroux proposed the use of silver membrane filters as they give a low background compared with other types. He also proposed the use of MoKα X-rays to minimize absorption effects.

It is recommended that the area of the diffraction peak should be measured for quantitative analysis (23). To enable this to be carried out on a large number of samples requires computer processing and equipment was therefore purchased with a computer-compatible output on punched paper tape.

The design of the Elliot Lake Laboratory dust sampling and assessment system for use in mines was developed during many studies over the past few years and is now named the Canadian Mining Personal Dust Sampling System — CAMPEDS.

Many major facets have to be taken into account and these are treated in separate chapters. However, many details and some ancillary studies are relegated to Appendices.

Accurate assessment of quartz in mine air involves both analysis and sampling, all aspects of which have to be considered.

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CHAPTER 2

OUTLINE OF DEVELOPMENT

The design of the Elliot Lake Laboratory dust sampling and assessment system, now called CAMPEDS, involved many separate studies. Advances in the design of the different components were made at various times and this chapter outlines that development.

The major emphasis at the start of the program in 1970 was in sensitivity for a quartz assessment and involved collecting as much quartz as possible on as small a filter as practicable. The major types of small battery operated gravimetric samplers with respirable size selectors were first compared with a horizontal elutriator in the laboratory dust chamber with the results given in Table 2.1.

These results suggested that Casella personal samplers would present much more quartz to the small X-ray beam than would any of the other samplers. It was also found there was considerable disagreement between the two U.S. coal mine samplers using the same size selector. This was found to be due to pulsations in the airflow given by the single acting diaphragm pumps used and has since been corrected by the manufacturers, thereby bringing the relative dust concentrations to 0.7 by the Cassella 113A. It should also be noted that with the Bendix, 25 to 50% of the dust collected was deposited on the internal surface of the cassette rather than on the filter where it could be analyzed.

The next stage of development was checking performance of the X-ray diffraction equipment on samples collected in a pure quartz dust cloud. These tests brought out four major observations:

1. quantities of quartz down to 0.25 μg in the X-ray beam could be detected by the X-ray system,
2. response was not linear, and apparently at the start of sampling some 30% of the quartz was not seen by the X-ray beam,
3. there was disagreement between the weight of quartz on the sample filter and that determined by the X-ray system when calibrated for evenly deposited quartz. This led to a complete calibration against dust collected on the filters,
4. a copper X-ray target was preferable to molybdenum.

This laboratory study was followed by a period of field work carried out with funding from the McIntyre Research Foundation. The major factors discovered during this period were:

5. quartz analysis could be carried out on most samples collected by the Casella personal sampler in mines,
6. interference by other minerals occurred fairly often and had to be allowed for,
7. in mines with diesel vehicles, clogging of the 25-mm diameter filters could occur in as little as 2 hours; however, as concentrations as indicated by weight loss on ashing, up to 10 mg/m^3 were found, it was hoped the sampling problem could be solved by controlling diesel exhaust rather than by increasing the filter area with the resultant loss in sensitivity for quartz,
8. as a personal sampler in mines two components of the Casella-plastic pump case was prone to damage, and the smoother needed

Table 2.1 - Dust collection characteristics of samplers

	Filter diam mm	Flowrate L/min	Relative respirable dust mass	mass/unit area	concentration
Casella MRE 113A	55	2.5	1	1	1
MSA coal mine sampler	37	2.0	0.27	0.7	0.34
Bendix coal mine sampler	37	2.0	0.41	1	0.51
Casella personal sampler	25	1.9	0.8	4	1

frequent repair; however, a metal cased version is now available.

After the system was proven in the field, studies have concentrated on the following considerations:

9. interference with quartz analysis,
10. absorbtion of X-rays in the dust layer,
11. methods of analyzing rock samples,
12. search for alternative filters,
13. X-ray diffraction analysis of varieties of free silica,
14. refinement of the X-ray diffraction technique for quartz analysis,
15. development of sampling head pump system for mounting on the miner's cap lamp battery,
16. development of a small combined impactor size-selector with a filter-cassette,
17. dust sources in mines as measured by gravimetric and quartz analysis,
18. the variation of full-shift personal samples preparatory to designing personal dust sampling programs.

Studies on consideration 9 to 16 have been completed and are dealt with either in the text or in the appendices; those on considerations 17 and 18 are incomplete and only preliminary results are given.

CHAPTER 3

QUARTZ ANALYSIS

Little difficulty was found in obtaining adequate sensitivity for X-ray diffraction assessment of quartz in the small quantities collected by portable battery operated dust samplers. However, many precautions are necessary to minimize errors and the major ones — permanent standards, calibration, interference, and absorption — are dealt with in the sections of this chapter which follow.

The detailed set-up of the equipment for optimizing high sensitivity quantitative analysis of quartz is given in Appendix A and for computer processing is giveg in Appendix C. Appendix D describes a method of preparing rock samples for analysis by this technique and Appendix E describes the ancillary studies carried out on varieties of free silica.

3.1. Permanent Quartz Standards

Leroux (1) in his development of the thin layer technique for X-ray diffraction analysis of quartz used the diffraction from an aluminum plate as an intensity standard. The diffraction angle is not the same as for quartz and is thus not easily used in conjunction with the automatic sample changer and step scanning. Therefore, permanent quartz samples were developed which ideally should simulate the dust deposit on the filters in particle size and surface density distribution.

In the first attempt, quartz samples taken on silver membranes were mounted on the automatic sample changer holders and sprayed with various lacquers. The samples were not sufficiently robust and the lacquer increased the background excessively.

The second method tried was to place a mixture of quartz and setting resin in a recess in a steel plate. This technique, although giving a high background -- mainly a very broad band about the cellulose d-spacing -- , was robust. A number of attempts were made before the standards were uniform across the area and the quartz sufficiently finely ground.

The procedure finally selected for preparing permanent standard quartz samples was as follows:

1. cut steel plate to fit automatic changer,
2. surface grind both sides,
3. turn 0.25 mm deep cylindrical depression the same diameter (22 mm)* as the dust deposit on the filter paper,
4. grind quartz (silica sand) in jet mill and collect in cloth bag,
5. disperse quartz in alcohol and let coarser dust settle,
6. mix suspension with resin and hardener and place in sample holder,
7. finish dust in resin and plate flat on surface grinder and reduce the layer thickness to about 0.012 mm.

These permanent quartz standards are not an ideal match in particle size distribution and surface density distribution for the Casella samples and are satisfactory as standards only if the X-ray diffraction arrangement is similar to that used in calibrating them.

Five quartz standards have been prepared, four for 25-mm diameter filters and 22-mm diameter dust deposits*, and one for 13-mm diameter filters and 10-mm diameter dust deposit. The four standards for 25-mm diameter filters were calibrated by comparing with the thicker samples collected in a pure quartz dust cloud and described in Section 3.2. and Appendix B. These comparisons were carried out 3 or 4 times on each of three strongest quartz diffraction lines, 1,0,0; 1,0,1; and 1,1,2. The repeat comparisons gave a standard deviation of about 4% in the estimate of the mass of quartz on each standard for each diffraction line. This variation is considerably greater than that, 0.5% calculated from counting statistics using the formula given by Klug and Alexander (2). Two factors contributing to this greater variation are the finite size and variability of position relative to the peak of each step, and variations in position of the limits of integration as chosen by the peak area computation program on a statistical basis.

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* Other changes have led to the dust deposits being 20.5 mm in diameter in Casella samplers and matching standards should be used.

In using these calibrations it is essential that the divergent slit be big enough for the beam to cover the full width of the standard and sample.

The use of permanent standards is essential, both for detecting malfunctions in the equipment and allowing for the long term decrease in X-ray tube output of about 30% in 2000 hours as shown in Table 3.1.

Table 3.1 - Variations in X-ray intensity with time

Date (1971)			X-ray intensity
July	16	a.m.	37034
	16	p.m.	38380
	19		38001
	20		37199
	21		36113
	22		32822
	23		31549
	26	a.m.	30463
	26	p.m.	30379
Sept.	7		33156
	8		35012
	9		32744
	10		33674
	23		32448
Nov.	5		31600
	10		31380
	15		28717
Feb.	10		28730
	11		27765
	22		27832
	23		27628
	23		27940
	25		28331
	25		27744

3.2. Calibration

The calibration of X-ray diffraction analysis of quartz was carried out by comparing X-ray intensity given by the standards with that given by samples collected in a pure quartz dust cloud. Detailed results are given in Appendix B. A number of major factors were found:

1. some quartz is lost in filter pores before dust bed builds up, resulting in shielding from the X-ray beam; this loss depends on the filter pore size and on size distribution of the quartz as well as of other dust; it is corrected by a thin sample correction factor;
2. absorption of X-rays in dust layer;
3. uneven deposition of dust on the filter -- heavier at the centre than at the edge is characteristic of many dust samplers;

Minor factors were also noted:

4. precise location of sample in the X-ray beam;
5. position of step with respect to peak centre;
6. variations in X-ray intensity over area of beam;
7. variations in X-ray intensity with time.

The effect of dust penetrating into the filter and of absorption is shown in outline in Fig. 3.1. together with the calibration, shown as a dotted line, obtained by taking the mean of the heavier samples. It can be seen that the dotted line is within 7% for all samples which have less than 20% absorption and small thin sample corrections and thus for most samples, adequate results are obtained without correcting for absorption. However, the dust losses on thin samples are appreciable and the use of the thin sample correction factor is desirable. The experimental derivation of the thin sample correction factor is difficult and may vary from batch to batch of filters. The best available values for silver membrane filters are given in Table 3.2., both for full effect and for correction to the mean calibration.

To minimize errors due to these factors it is essential to calibrate against dust samples

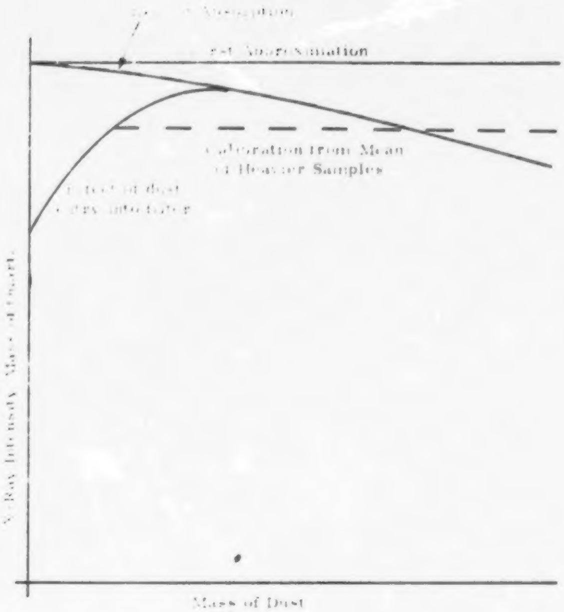

Fig. 3.1 - Schematic of effects of absorption and penetration of dust into filter at start of sampling.

Table 3.2 - Thin sample correction factors

Sample weight mg	Thin sample correction factor		
	Full effect	Correction to mean calibration	
0.0	1.5	1.4	1.16*
0.05	1.42	1.33	1.13*
0.10	1.35	1.26	1.19*
0.15	1.30	1.21	1.07*
0.2	1.24	1.15	1.05*
0.3	1.16	1.08	--
0.4	1.10	--	--
0.5	1.05	--	--

*Estimated values for 3-μm pore size silver membrane filters. Others are experimental values for 5-μm pore size.

prepared by a method as similar as possible to the field technique and to use "standard" samples that are as close a match to field samples as practical. Although it has been possible to produce reasonable samples for calibration using the appropriate dust sampler in a pure quartz dust cloud in the laboratory test chamber, the standards match the filter in diameter but not in uniformity of deposition.

A small zero error was observed when comparing measurements of pure quartz samples made on the four lines using cobalt radiation. The origin of this zero error appears to be in the calculation of area using a linear approximation for the background pattern from the silver filter, whereas this background is curved or irregular, particularly for the 1,0,0 and 2,1,1 lines. This zero error amounts to adding approximately 10 μg to estimates on the 1,0,0 line and 4 μg to those on the 2,1,1 line. More work is required to explain this problem.

3.3. Interference

A large number of minerals produce X-ray diffraction (XRD) patterns in which one or more lines are similar or quite similar to quartz. For instance, the common mineral muscovite has a strong line at a crystal lattice spacing of 3.32A, close to the strongest quartz line at 3.34A and interference with quartz analysis occurs.

Figure 3.2 shows two of the conditions that can occur with two close lines. In the first, Form 1, the two lines merge and appear as a single slightly wider line, and in the second, Form 2, the peaks are clearly resolved. When computing the area under the peak assuming a linear background, the hatched area would be omitted.

A number of approaches towards avoiding or correcting for interference are possible — the following have been examined:

1. identifying minerals present in the ore from mine geologists' data and choosing a quartz line that is clear of interference,
2. XRD scanning of ore and airborne dust samples from the mine and identifying minerals present and choosing a quartz line that is clear of interference,
3. visual checking of the XRD scan of each sample for adjacent peaks and departure

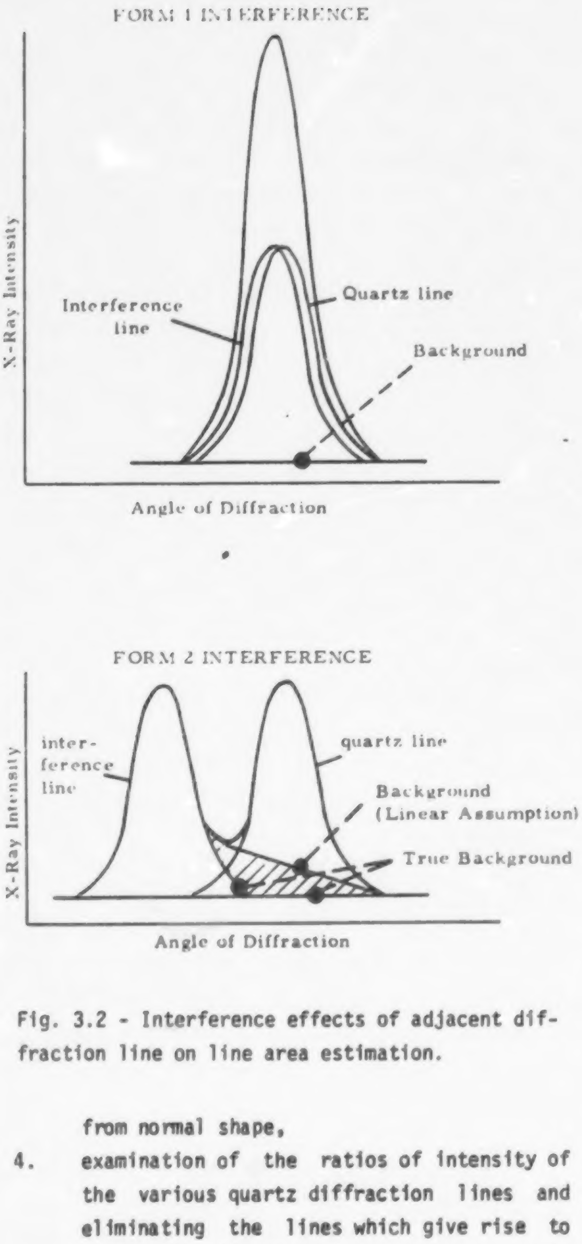

Fig. 3.2 - Interference effects of adjacent diffraction line on line area estimation.

from normal shape,

4. examination of the ratios of intensity of the various quartz diffraction lines and eliminating the lines which give rise to ratios markedly different from pure quartz samples,
5. comparing estimates of quartz on each sample obtained by using a number of diffraction lines,
6. computer analysis of each scan using curve-fitting with least squares criterion.

The first approach entailed obtaining mineralogical reports from the mine geologists. However, these reports were not adequate to determine the XRD patterns using standard references for many minerals, particularly those with variable composition such as the plagioclases (3,4), it was thus necessary to confirm the patterns using the second approach. In this approach a number of samples of ore and host rock were obtained from each mine, crushed and scanned by XRD. This was successful in some cases where the number of minerals present in significant quantity was small. For the Elliot Lake uranium mines the only interference found was muscovite (sericite) on the 1,0,1, quartz line. The analysis could not be completed for mines with a large number of minerals present in significant quantity, such as the Timmins gold mines. For these, even after identification and confirmation of seven minerals in the XRD pattern, some medium strength lines were not allocated to minerals.

The third approach is useful in detecting Form 2 errors and, incidentally, in detecting samples which become misplaced in the automatic sample changer, but for only one other sample has useful information been gained from the shape of the diffraction line. When a Form 2 error is recognized, the quartz diffraction line may be expected to provide a low estimate of the quartz content and so the reliability of the result should be discounted.

In investigating the fourth approach, the ratios of intensities of three quartz diffraction lines were measured. The range of results obtained from four types of dust samples are shown in Table 3.3.

Table 3.3 shows that only in the case of uranium mines are the results compatible with the assumption that there is no significant interference on two of the lines — the 1,0,0 and the 1,1,2. The range of ratios for the other mines for all three comparisons are such that no conclusions can be drawn.

In the fifth approach, four estimates of the quartz in each sample are made using the four strongest diffraction lines of quartz that are clear of the diffraction pattern of the filter material (silver metal). The results obtained

Table 3.3 - Ratio of intensities of quartz diffraction lines

Airborne dust samples	Range of intensity ratios (Integrated line areas)		
	1,0,0/1,0,1	1,1,2/1,0,1	1,1,2/1,0,0
Pure quartz	0.20-0.22	0.083-0.10	0.4 -0.44
Uranium mines	0.16-0.21	0.06 -0.10	0.4 -0.45
Gold mine	0.05-0.60	0.15 -0.18	0.17-1.9
Nickel mine	0.12-0.38	0.05 -0.12	0.23-0.5

from a representative range of samples are shown in Table 3.4. It can be seen there is reasonable agreement between the estimates in many cases, but in others they differ by as much as 300%. Form 2 errors were found on some lines on certain samples. It is assumed that as Form 1 errors are not observable and give rise to high estimates of quartz, the best estimate is given by the lowest one that does not show Form 2 errors.

The sixth approach — form fitting with least squares criterion using a computer — has been used successfully for separating overlapping lines in many similar problems, for instance in gas chromatography (5). However, in the case in question, with diffraction lines that are weak with respect to background and are distorted due to bent filters, the preliminary examination suggested that long counting times and extended scanning widths would be necessary for a successful analysis. This approach does not seem as promising as producing multiple estimates of quartz from different diffraction lines — approach 5 — and has not been investigated further.

It is clear that interference is a problem in quartz analysis and that considerable effort could be spent in checking each sample and it is thus necessary to consider the object of dust sampling and quartz analysis in mines. The requirement in most sampling is to demonstrate that the dust level is below a legally defined limit and all samples with a lower level than this and free of Form 2 errors can be reported as having a maximum rather than an estimated quartz content. Form 2 errors can be detected on the computer print-out by:

1. an adjacent peak,
2. a marked change in background or background slope from similar samples, or
3. a marked disagreement between estimates of quartz by peak height and by line area.

Samples with Form 2 errors or quartz content above the threshold limit must be analyzed on a second line to determine the maximum content or to confirm the high estimate. It may be necessary to extend this analysis to a third or fourth line for some mines.

Ideally, the quartz estimate should be based on a weighted mean of measurements on the various lines allowing for their different accuracies and for indications of interference. In many mines with low dust concentrations, the disagreement between lines appears more often to be due to errors than to interference, and thus if one uses only lines not prone to interference the simplest process is to average the measurements obtained.

In the mine samples analyzed to date it appears that the 1,1,2 quartz line is the least subject to interference and most suitable for the first estimate. However, the 1,0,1 line has eight times the intensity, a slightly greater sensitivity, and is more suitable in some mines. It is thus a close choice between these two lines for obtaining the first estimate. The 1,0,0 line is least sensitive because of the higher background at the lower angle and to date has appeared to be the least useful of the four lines examined.

Interference has frequently been found and appears to be a disadvantage of the X-ray diffraction method. However, in most cases interference has been observed only because of disagreement between the independent estimates. The chemical method — separating silicates from free silica by dissolving in hot phosphoric acid — can provide only a single answer, and the variable dissolution rates of silicates and free silica with type of mineral and particle size give rise

Table 3.4 - Comparison of quartz analyses using some of the diffraction lines available

Mine	Mass of quartz estimated in mg using the diffraction lines indicated							
	1,0,0		1,0,1		1,1,2		2,1,1	
	Height	Area	Height	Area	Height	Area	Height	Area
A			.017	.019	.025	.024		
			.030	.031	--	--		
				.014		.015		.001
				.022		.01		.022
		.01		.01		.06		.01
B	.023	.049	.006	.007	.007	.002	.010	.003
	.110	.093	.20	.305	.175	.15	.185	.165
	.185[1]	.160[1]	.064	.077	.057	.053	.050	.041
C				.24		.00		
				.32		.27		
D		.86		.45		.24[1]		.60
		.30		.24		.11[1]		.26
E[2]		1.03		.83		.36[1]	.81	1.16
F		.32		.04		.02		
				.05		.03		.04
G				.06		.12		.04
H		3.14		.26		.15		.21
H'		.24		.21		.16		.14
H'[3]		.64		.15		.13		.09

[1] Adjacent peak detected.

[2] Percentage of quartz in the crushed ore determined by X-ray diffraction: i.e., 24%, 64%, 15%, and 35% respectively for each of the four lines. These figures compare with the 30-40% found by other techniques.

[3] The one sample H' from a coal mine was ashed and reanalyzed with the results given in this line.

to unknown error. Infrared absorption can provide three semi-independent estimates of free-silica content — one pair is a doublet, and serious interference has been reported (6).

3.4. Sample Absorption Studies

'Thin' samples are used in quartz analysis to avoid the need to determine corrections for matrix and absorption effects and non-linear calibrations. Although molybdenum radiation, because of its high energy and low absorption, would permit measuring thicker layers, it has very great disadvantages in sensitivity and resolution compared with longer wavelengths.

It is expected that as the thickness of a sample increases, the ratio of intensity of the diffracted peak to the mass will drop due to absorption. This decrease in ratio can be given for small losses simply as:*

$$\text{Ln}\ (R_o/R_m) = \mu_m\ m\ \text{cosec}\ \theta$$

where: R_o = ratio of intensity to mass extrapolated to zero mass;

R_m = ratio of intensity to mass at mass m;

m = mass of dust per unit area;

μ_m = mass absorption coefficient for X-rays;

θ = Bragg angle.

This equation assumes that on the average the X-rays pass through half the depth of the dust layer twice. For the silver peaks, X-rays pass through the dust layer twice and a factor of 2 is added to the right-hand side of the equation.

Using the values for mass absorption given in the X-ray handbook (7) mass limits for selected minerals can be calculated corresponding to R_m 20% less than R_o due to absorption. The selected minerals are: SiO_2, representative of silicates and quartz; FeS_2 representative of iron, the commonest mineral-forming element that is a heavy absorber of copper radiation; and $CaCO_3$ representative of calcium, the commonest element that is a heavy absorber for cobalt, iron, and chromium radiations. Table 3.5 shows that mass limits for each radiation for each of the four lines of quartz together with that for the strongest silver line from the supporting filter. It can be seen that the mass limits decrease generally in the order from Mo to Cr.

For 25-mm diameter filters with an effective area of 3.5 cm² the absorption mass limit for molybdenum radiation is well above the threshold limit value for all dusts, while with copper radiation, the element iron is a limiting factor. With chromium radiation, the quantities of mineral-forming elements — potassium and calcium — are limited to 1.4 mg on the filter for analysis by the quartz 1,0,1 diffraction line. Considerations of elemental abundancies in the earth's crust show that of the elements which could be problems with these characteristic radiations — iron at 5%, calcium at 3.6%, and potassium at 2.6% — are the most important. From absorption characteristics, on average, cobalt and iron radiation might well be preferable to copper because they have less absorption in the common rock-forming element, iron. Chromium radiation would seem more likely to give rise to absorption problems than iron radiation.

It should be possible to check absorption by measuring the intensity of the silver 1,1,1 line through the dust deposit. Figure 3.3 shows a plot of this intensity, as peak height, against mass of some samples using copper radiation. It can be seen that with pyrite samples the loss in intensity with mass is about half that predicted (Table 3.5). Figure 3.4 shows the effect of sample thickness on X-ray intensity mass ratio of the four quartz lines for the mixed pyrite-quartz samples shown in Fig. 3.3. The fall in intensity with thickness is less than expected from absorption of the silver line shown in Fig. 3.3 and from comparisons between quartz and silver lines shown in Table 3.5.

With field samples, distortion of the filters sometimes leads to line broadening and reduction in peak height and it is necessary to determine peak area. This has been done by multiplying net peak height by half the width on the recorder trace with the results shown in Fig. 3.5 for all field samples received in a given period using Cu radiation. Figure 3.6 shows some of these repeated using Cr radiation. It can be seen that the variation in silver peak

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* J. Leroux (8) has given a more rigorous treatment of absorption in dust samples which is necessary for thicker samples than considered here.

Table 3.5 - Calculated mass limits for 20% departure from linear calibration with a selection of materials and radiations

Substance	Diffraction	Mass limit mg/cm^2 for radiations from				
	line	Mo	Cu	Co	Fe	Cr
SiO_2	Q 1,0,0	5.3	1.7	1.1	0.77	0.63
	Q 1,0,1	6.8	2.1	1.4	1.0	0.82
	Q 1,1,2	12	3.9	2.5	1.8	1.5
	Q 2,1,1	14	4.6	3.0	2.1	1.7
	Ag1,1,1	4.8	1.5	1.0	0.7	0.58
$CaCO_3$	Q 1,0,0	2.6	0.62	0.44	0.35	0.28
	Q 1,0,1	3.4	0.78	0.56	0.45	0.37
	Q 1,1,2	6.0	1.45	1.05	0.85	0.67
	Q 2,1,1	7.0	1.7	1.2	1.0	0.75
	Ag1,1,1	2.4	0.56	0.4	0.32	0.26
FeS_2	Q 1,0,0	0.85	0.22	0.50	0.39	0.31
	Q 1,0,1	1.1	0.27	0.6	0.5	0.41
	Q 1,1,2	2.0	0.5	1.4	0.95	0.76
	Q 2,1,1	2.3	0.6	1.5	1.1	0.85
	Ag1,1,1	0.75	0.2	0.45	0.35	0.29
Oxygen[1]	Q 1,0,0	30	10	7	5	5

[1] This is entered as an ultimate possible low mass for organic compounds (combustible dust).

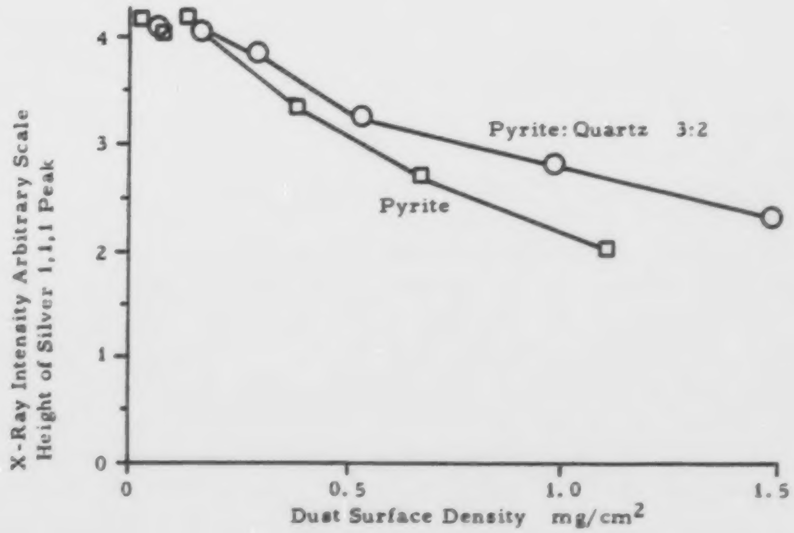

Fig. 3.3 - Absorption of CuKα X-rays in dust layer as measured by intensity of silver 1,1,1 peak.

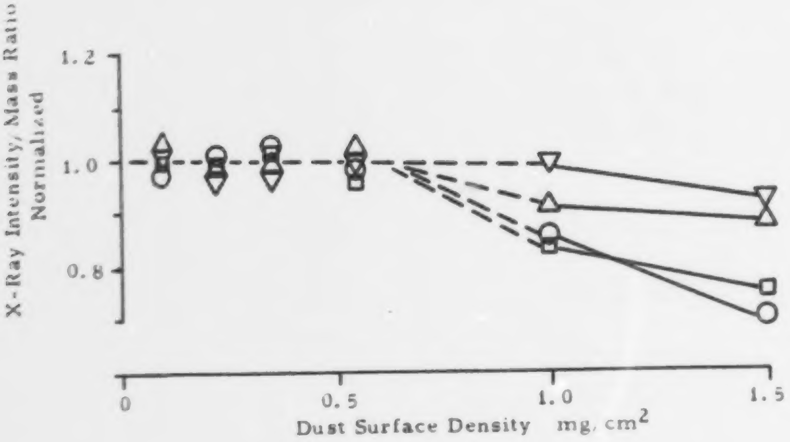

Fig. 3.4 - Effect of absorption on intensity of quartz peaks in 3.2 pyrite-quartz dust deposit.

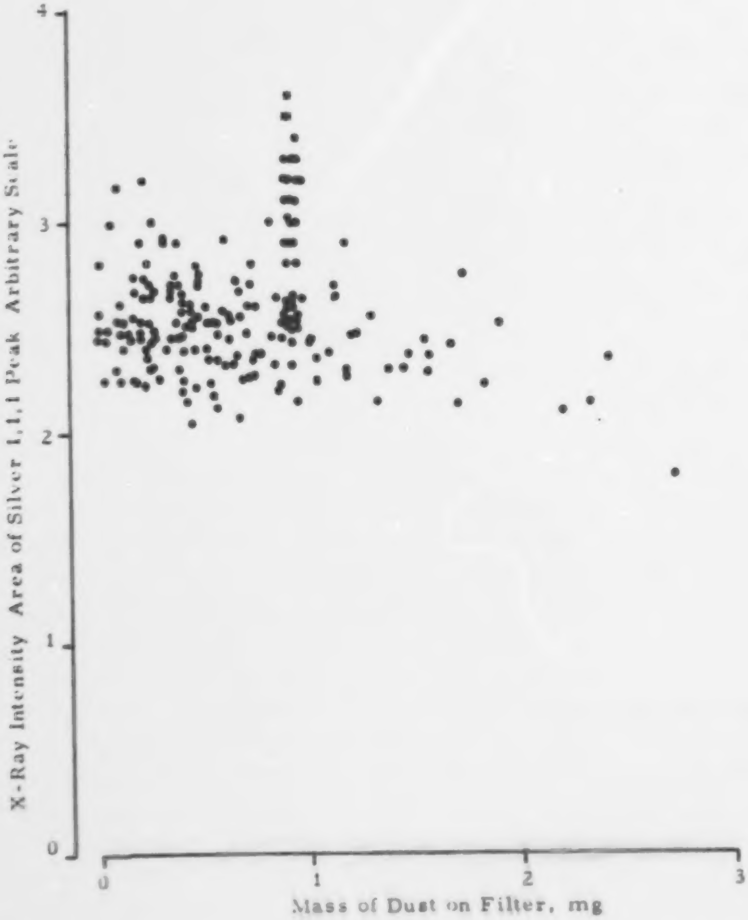

Fig. 3.5 - Intensity of silver peak measured through dust deposit on field samples — copper radiation.

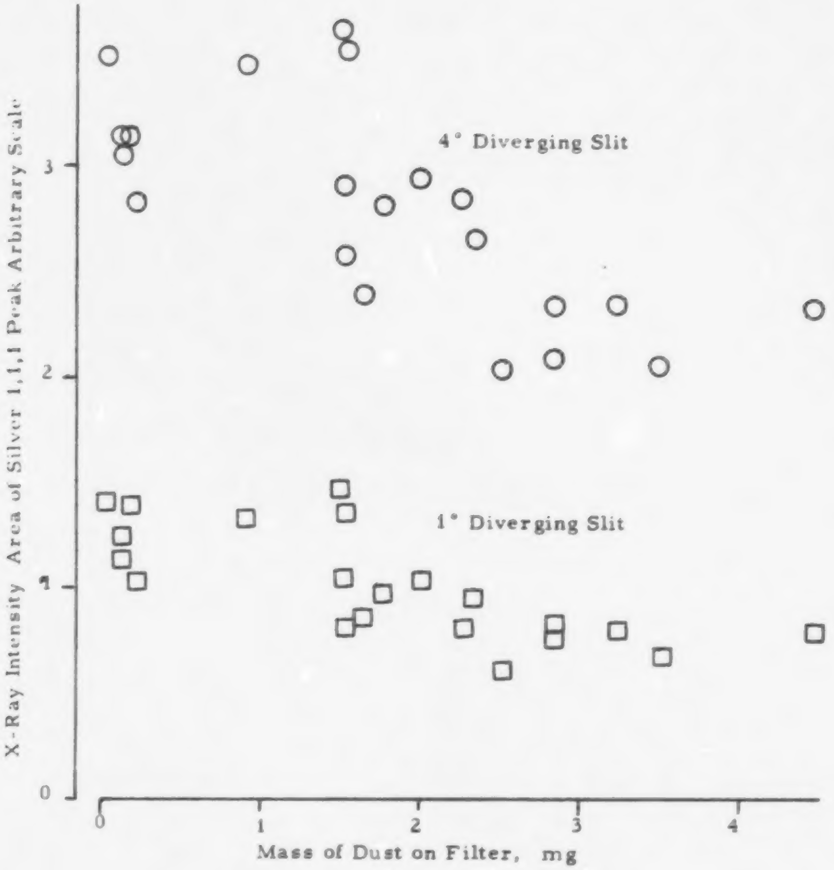

Fig. 3.6 - Intensity of silver peak measured through dust deposit on field samples — chromium radiation.

intensity is much greater than the possible effect of absorption for most of these samples, and one cannot be sure that there is significant absorption until the peak height is less than 2 for Cu radiation or less than 2.5 for Cr; the absorption loss could then be as high as 50% or as low as 10%.

These results were obtained with a wide slit and it is possible that the edges of the filter outside the dust deposit caused errors in estimating absorption of the dust layer. Also the centre of the dust deposit is heavier than the edges. Figure 3.6 shows intensity of the silver peak through the dust deposit when illuminated over two areas, 26 x 12 and 10.5 x 12 mm. This suggests that the relative absorption is about twice as great in the central portion of the sample as at the edges. In these experiments the second type of filter holder was used and a 22-mm diameter dust deposit was exposed through a 23-mm diameter orifice with some clear silver showing. This does not explain most of the variations found in field samples and it is assumed the main reason is the 'tarnishing' of the silver metal leaving a highly X-ray absorbent silver compound on the surface. Leroux has suggested that intensity of the silver peak from the back of the filter should be compared with that from the front through the dust deposit as a measure of absorption in the dust layer (8). Although this procedure corrects the batch-to-batch variation found for clean filters, it gave little improvement on the single dust-side measurement when applied to field samples, and as well, it is easier to use the samples with little dust as a control for the heavier. A significant factor appears to be preferential tarnishing on the dust side, either during collection or by reaction with the deposited dust. It is known that hydrogen sulphide, a not uncommon gas, reacts with silver metal to produce silver sulphide, which has occasionally been detected by XRD.

It is concluded that errors greater than 20% are only likely to occur in a few airborne dust samples even if chromium radiation is used. Gross errors greater than 50% can probably be detected and corrected by measuring intensity of the silver diffraction peak through the dust layer using the relative masses given in Table 3.5. Observing intensity of the silver peaks should be carried out as a routine on heavier samples until such time as further experience indicates if it is worthwhile.

Cobalt-target X-ray tubes can be obtained in a high intensity version. The absorption by silicon, calcium, and potassium minerals would be intermediate to those for Cu and Fe radiations, although the absorption by iron-bearing minerals would be less and is recommended as the best compromise.

3.5. Summary of Quartz Analysis

Permanent standards must be used to avoid effects of changes in X-ray intensity with time and setting. Although any standard substance could be used, it is simpler to use quartz of at least the same diameter as the dust samples as it will minimize errors due to changes in beam angles and can be processed with the same scanning limits and computer program. Internal standards with transfer to a second filter have been used by some analysts but although this overcomes matrix errors, it appears to increase the possibility of error due to interference (9).

Calibration using samples collected in a pure quartz dust cloud appears desirable in direct analysis on the collecting filter to minimize errors from uneven deposition and to collection of dust in the body of the filter where it is hidden from the X-ray beam. Because dust collected in the body of a filter is difficult to remove it is possible that this effect also occurs in transfer techniques and that calibration of the whole process is desirable.

The deposition of dust in the body of the filter at the start of sampling before the dust bed builds up has been handled by the use of a thin sample correction factor determined during calibration in a pure quartz dust cloud. Clearly the deposition and the factors depend both on the pore size of the filter and on the size distribution of the dust. The results obtained suggest that the factors would differ from 1 by a factor of about 2 for 5-μm pore size compared with 3-μm pore size silver membrane filters and thus there might be a slight batch to batch variation due to changes in average pore size. The effect of particle size distribution on dust bed build-up and penetration into the filter might be significant for mineral dusts compared with the much finer diesel exhaust particulates. However, as it is very difficult to estimate and to carry out experiments, the assumption is made that the thin sample correction factors for the weight of quartz on the filter determined in pure quartz dust are applicable to other samples if the weight of total respirable dust is substituted for the weight of quartz. Thus, build-up of the dust bed and its penetration by quartz bears the same relation to its total weight as does that of a pure quartz dust bed.

Because determination of the thin sample correction factors is difficult, possibly not too accurate, and does not introduce substantial errors in samples above half the suggested target values, it is suggested that the values given in Table 3.2. can be used without excessive error for all silver membrane filters.

Interference, as indicated by disagreement between the estimates using the four diffraction lines of quartz, occurs frequently and would appear to be a disadvantage of the X-ray method; however, there is no such easy method of checking for interference in the other methods — chemical and infrared — and minor interference would therefore not be detected. However, adjacent peaks and the resultant low value for quartz are readily detectable and, except in these cases, one can take the measured value as an upper limit for quartz in the sample. Consideration should be given to carrying only confirmatory analyses on a second diffraction line for the heavier samples.

The results obtained suggest that absorption or matrix effects are not significant for most field samples that do not exceed the suggested target value of 2 mg/m³ (2 mg per filter) of total respirable dust, and thus are not important. However, the use of a cobalt target X-ray tube gives rise to least absorption by the commoner mineral-forming elements and still having the longer wavelength desirable for increasing resolution, minimizing interference, and giving a high intensity.

Computation of peak area is desirable to minimize effects of distorted filters broadening the diffraction lines. The program used defines the background areas to avoid adjacent peaks. It appears probable that the use by many programs of fixed positions for counting background increases errors due to interference and curved background on very thin samples, found to have a width of less than one third of that for heavy samples.

The advantage of a high count rate obtained by running the X-ray diffraction equipment with the maximum slit widths normally supplied outweighs the disadvantage of the resultant slight reduction in resolution. The graphite monochromator in the diffracted beam has the advantage of decreasing the background, particularly at high diffraction angles and on field samples, as well as increasing the intensity compared with filters to remove the Kβ radiation.

The emphasis in the development of the X-ray analysis technique has been on the rapid processing of large numbers of samples rather than on the accurate analysis of individual samples. Field results have shown good reproducibility of side by side samples ± 15%, but very great variations of up to 10:1, between the highest and lowest full shift samples taken with one man nominally doing the same job in the same working place on different days. Duplicate analyses on one line, either 1,0,1 or 1,1,2, suggest that the accuracy of quartz analysis can be better than ± 5 μg or 10% whichever is the greater. Larger errors can creep in due to interference, filter damage, transmission and transcription. It is believed that if simple precautions are taken, only a few single analyses will have errors greater than 25%. These precautions are:

1. examining filters for damage, estimating correction factor for section of cake missing;
2. checking interference on heavier samples from each mine, choosing one line least subject to interference;
3. checking the printout on each sample for an unusual background slope and disagreement between peak height and peak area estimates of quartz, repeating analysis or computation.

Because of variations in dust concentration, better results would be obtained by taking multiple samples and doing single analyses rather than by spending the same effort in doing multiple analyses of single samples. For critical work on undamaged samples, greater accuracy can be obtained by performing multiple analyses with rotation of samples in the holder.

3.6. References

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CHAPTER 4

FILTER CHARACTERISTICS

The filter is required to collect a sample of dust from the air, to support it in the X-ray beam for analysis, and to enable it to be weighed before and after ashing. Clearly, a number of factors affecting these processes must be considered:

1. collection efficiency,
2. site of dust deposition on the surface or in the body of the material,
3. airflow and pressure drop,
4. shape and size,
5. background for X-ray diffraction analysis,
6. hygroscopicity,
7. ashing.

4.1. Dust Collection Efficiency

A sensitive method is readily available in the Elliot Lake Laboratory for determining the passage of airborne quartz through filters by estimating the quartz deposited on a back-up filter of fine pore size using X-ray diffraction.

This method was applied to a number of filter materials of 25-mm diameter with nominal pore sizes of 3 to 8 µm by sampling fine quartz dust clouds (80% respirable) produced by a fluid energy mill from silica sand (Ottawa, Illinois) (1). Silver membrane filters of 0.8-µm pore-size were used as backup filters. The experiments were carried out in two ways: firstly, by making single runs in which 0.8 mg of dust was collected on the first filter at a flow rate of 1 L/min; and secondly, by making a run in which the first filter was changed three times while a total of about 1 mg of dust was collected. The quantities of quartz found on the second filter are shown in Table 4.1 to be, with one exception, the indicated amounts on the backup filter of 1 to 5 µg, which is at the lower limit of detection for quartz by X-ray diffraction and corresponds to less than 1% penetration of dust through the filters. The one exception with 58 µg on the filter was not confirmed by a replicate run and was assumed to be due to contamination during handling. This suggests that the other measurements may also have overestimated the penetration. Recent work has shown that 40 to 70% of 0.3-µm DOP aerosol penetrates the 5-µm pore size silver membrane filters, while 2 to 5% penetrates the 3-µm pore size (2). Even so, it is concluded that all these filters collect respirable mine dusts adequately, and that the amount of quartz penetrating the silver membrane filters is much less than the amount corresponding to the light-sample correction factor as shown in Appendix B. This implies that the light-sample correction is due to entry of the quartz into the body of the filter at the start of sampling and its shielding from the X-ray beam by the highly absorbent silver.

It is assumed that all the finer pore sizes have a higher collection efficiency than the coarse ones listed.

4.2. Site of Dust Deposition

Even though all the dust is collected by the filter, there is a possibility that some of it may enter the pores where it is partially or completely shielded from the X-ray beam by the filter material. The significance of this depends both on the degree to which dust enters the pores and absorption of X-rays by the filter material. The effect has only been examined for silver membrane filters. In the original calibration (Appendix B) it was found there was a reduction in the X-ray intensity/mass ratio for very light samples. This was explained by fine dust penetrating through or entering into the body of the filter prior to the dust bed building up. Once a dust bed is formed it can be expected that no dust will penetrate it. The effect is given in detail in the section on calibration and the dependence of this effect on the pore size of silver membrane filters is outlined in Table 4.2.

It can be seen that the X-ray intensity/mass ratio is less for light dust deposits than for the heavy for the three larger pore sizes but is slightly greater for the 0.8-µm pore size. This last relationship is explained approximately

Table 4.1 - Dust passing through filter materials

Filter	Pore size µm	Quartz on backup filter, µg 1st test	2nd test
Flotronics	5	1	30
Flotronics	3	2	2
Metricel	5	2	2
Versapor	5	4	5
Millipore	5	2	3
Nucleopore	5	2	5
Sartorius	8	2	2
Acropor	3	2	5
GF/A	5	2	2
GF/E	5	4	2

Table 4.2 - Dependence of body deposition of dust on filter pore size

Pore size	Ratio X-ray intensity to mass	
µm	400-500 µg dust	30-50 µg dust
5	146	101
3	142	125
1.2	157	135
0.8	154	158

by the expected effect of absorption in the quartz (Section 3.4).

4.3 Airflow and Pressure Drop

The airflow through and the pressure drop across a filter varies greatly with pore size and much less markedly with type. It has been found that with filters of pore size of 3 µm or larger the main limiting factors on the thickness of a dust bed have been the suction available from the pump, the airflow per unit area, and resistance of the dust bed. With smaller pore sizes than this, the higher resistance of the filter itself leads to a decrease in the attainable thickness of the dust bed. Five µm pore size filters have been used in most of the field measurements.

4.4. Shape and Size of Dust Deposit

Clearly, sensitivity depends on the mass of dust in the X-ray beam and thus on the shape and size of the dust deposit as well as on its thickness. As has been shown, the thickness of the dust deposit is limited by its resistance to airflow. Thus, if other things – sampling time, air velocity normal to the filter surface and concentration – are taken as constant, the mass of dust in the beam is proportional to the area of dust deposit and reaches a maximum when this area is equal to that of the beam.

If dimensions of the dust deposit were made equal to those of the beam, the accuracy would depend on alignment, but not on the uniformity of the dust deposit. With a larger deposit the dependence on accuracy of alignment decreases and the uniformity of deposition becomes important. For deposits smaller than the beam, neither alignment nor uniformity of deposition are important, but sensitivity is less.

The projection of the beam on the sample is normally rectangular, but rectangular filter holders could only reasonably be made in large numbers from moulded plastic. Some prototype metal units have been made and used successfully. The compromise of using circular filters of a diameter less than or equal to that of the beam has been used on some laboratory apparatus. For field use this would result in the flow rate being reduced to 1 L/min with available battery-operated portable pumps, thus rendering the gravimetric assessment less sensitive.

The other compromise of using circular filters larger in diameter than the beam has been used in most cases. It is found that most dust samplers do not give a uniform dust deposition and that the calibration technique must allow for this. Dust samplers give errors of up to 40% if the X-ray is calibrated with filters having uniform dust deposits.

Considering all these factors it has been concluded that the possible merits in using filters smaller than 25 mm diameter in dust samplers intended for underground use do not justify the considerable expense involved in changing the available dust-sampler pumps and size selectors. However, if a dust sampler were to be designed there could be merit in choosing a rectangular deposit about 1 mm less in size than the beam and choosing a flow rate to suit.

4.5. Background for X-ray Diffraction Analysis

It has been shown that quartz determination in airborne dust can be carried out directly on dust samples collected on filter papers. Silver membrane filters have been recommended (3) as the most suitable for this purpose. The suitability of a filter lies in its providing a low even background of scattered or diffracted X-rays at the positions of the quartz lines. The diffraction patterns from the six types of clean filter examined using CuKα X-rays are shown in Fig. 4.1. Similar results were obtained using CrKα X-rays.

Assuming a linear background in the neighbourhood of the diffraction line, the sensitivity for determination of quartz can be calculated from the formula given by Klug and Alexander (4):

$$P = 0.675\ (N_T + N_B)^{\frac{1}{2}}\ (N_T - N_B)^{-1}$$

where P is the relative probable error, N_T is the total count (including the background count $-N_B$) adapted approximately for the step scanning assessment of the area under the line used in the Elliot Lake Laboratory.

For the sensitivity limit in the step scanning (0.04°2θ per step was used), let P be 0.5, then:

$$MT^{\frac{1}{2}} = (1.8\ M/A + 25\ B/A)^{\frac{1}{2}}$$

where M is the sensitivity limit in μg of quartz,
T is the counting time per step in seconds,
A is the area of the diffraction peak per unit mass of quartz (counts $sec^{-1}\ \mu g^{-1}$); and
B is the background (counts sec^{-1}).

Note, on very light samples the peak is very narrow and the background to peak count ratio gets very large.

It has been found that the maximum useful counting time per step is limited to 100 seconds by small random 'diffraction' peaks on the silver membrane filters and assuming the same counting time limit is found for other filters the calculated sensitivity limits are given in Table 4.3 together with other relevant data for the stronger independent quartz diffraction lines using CuKα radiation.

It is necessary to analyze for quartz using all available independent diffraction lines because of interference by other minerals. It can be seen that silver membrane filters are most sensitive and have four lines available and that nucleopore filters (1), with five lines available, are almost as sensitive. The other filters are less sensitive and, on the original traces, also show more random small peaks than do silver or nucleopore filters, which would decrease the maximum useful counting time per step, and therefore the sensitivity.

4.6. Hygroscopicity of Filter Material

Filter materials which absorb water require carefully controlled atmospheric humidities prior to and during weighing, which greatly increases the time and effort required. Thus low

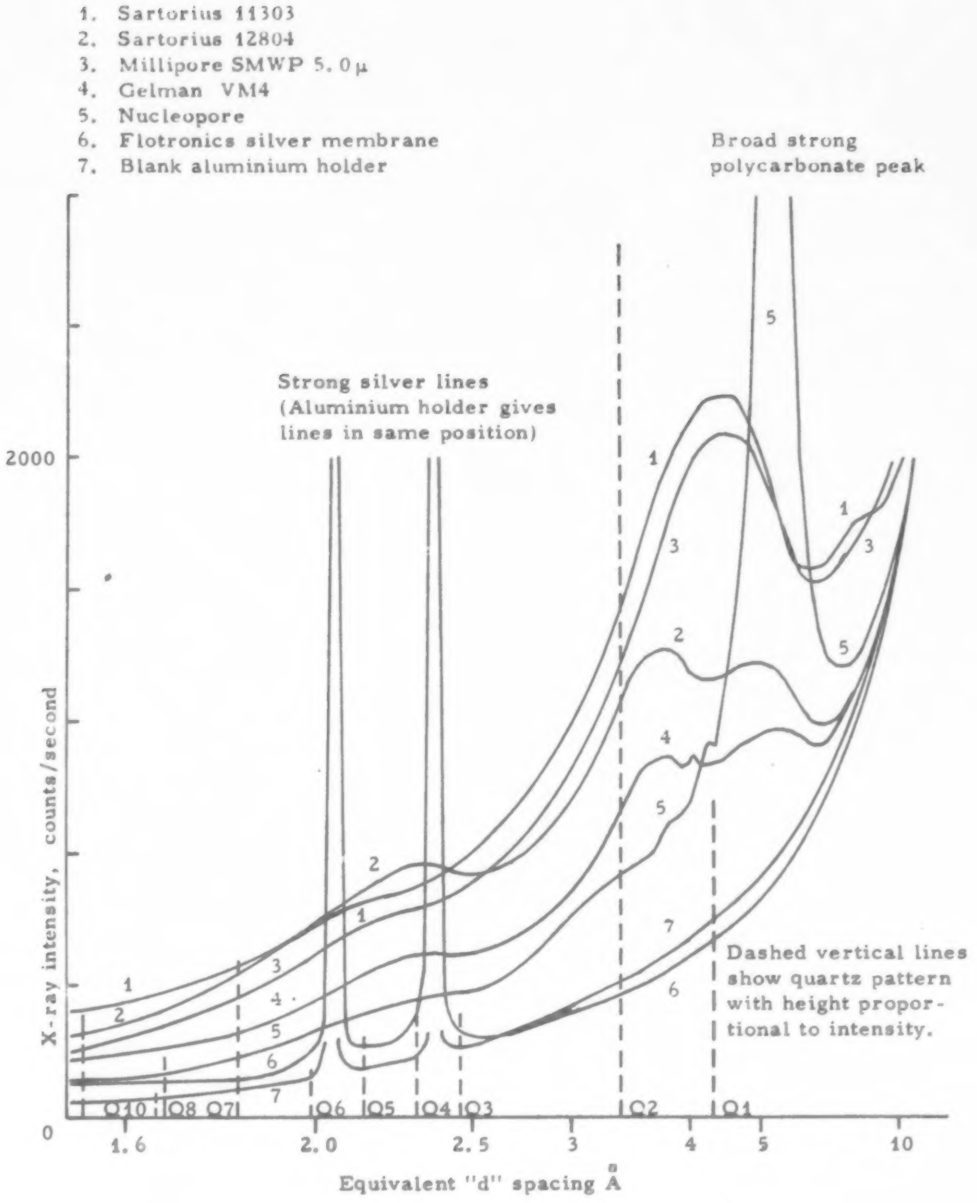

Fig. 4.1 - X-ray diffraction patterns from clean filters.

water absorption is desirable and is claimed by the manufacturers for many types of filter material. Experience with glass fibre, vinyl, silver and polycarbonate filters have confirmed this and drying to a specific relative humidity is not required. However, moisture will deposit in the pores at high humidities and all filters must be conditioned and weighed at relative humidities below 80%. The Elliot Lake Laboratory uses a domestic type dehumidifier in the balance room to achieve this in humid weather.

4.7. Ashing

Ashing of the dust samples is desirable

Table 4.3 - Characteristics of filter papers relevant to quartz analysis for the six strongest quartz lines

	Quartz lines					
Filter type[1]	Q1 100	Q2 101	Q3 110	Q4 102	Q7 112	Q10 211
X-ray intensity[2] of quartz peaks in counts sec^{-1} μg^{-1}						
	5.5	28	3.3	3.3	4.5	2.2
Background from filters in counts sec^{-1}						
1	2150	1500	800	700	450	320
2	1300	1250	720	750	430	255
3	2000	1350	700	600	360	205
4	1050	900	500	500	250	180
5	1100	700	370	350	175	110
6	500	350	270	300	105	110
Sensitivity for quartz determination in µg for 100 second counting time per step						
1	NLB[3]	NLB	8	7.3	5	6
2	NLB	NLB	7.4	7.5	5	5
3	9.5	NLB	7.3	6.7	4.5	5
4	6	NLB	6	6	3.5	4.5
5	NLB	2.5	5.3	5	3	3.5
6	5	1.8	NLB	NLB	2.5	3.5

(1) See captions on Fig. 4.1.

(2) Given as peak area with a step size of $0.04^{o}2\theta$

(3) NLB - Not usable except for very large quantities of quartz because the background is not linear.

to determine the mineral and combustible portions separately and to identify the significance of their respective dust sources. Silver filters can be ashed at temperatures between 480 and 500°C with very little distortion of the filter or disturbance of the mineral dust layer. The change in tare weight is less than 0.03 mg. Nucleopore filters can be ashed completely, leaving a negligible ash from the filter; however, the dust layer is loose in a dish and needs further handling for X-ray analysis.

4.8. Choice of Filter

To summarize, it is believed that silver membrane filters are the most suitable for the gravimetric — X-ray diffraction dust sampling system because of robustness, negligible weight change on ashing, and low background for X-ray diffraction, and are worth somewhat greater cost.

Nucleopore filters are difficult to handle and readily lose a dust cake. They could be more suitable than silver, however, if other analyses were required such as by infrared absorption. Their handling and dust retention might be improved by mounting and handling them in a rigid clip such as is used on the Casella 113a gravimetric dust sampler (5).

Consideration is being given to the use of quartz analysis using relatively cheap infrared absorption equipment at the mine with confirmation on the heavier samples at a central laboratory using X-ray diffraction. Preliminary results suggest that Gelman DM 5000 filters are sufficiently transparent to infrared and have background suitable for XRD analysis of quartz down to less than 50 µg.

4.9. References

1. Filter material identification and manufacturers: Flotronics silver membrane FM-5 — Selas Flotronics, Spring House, Pa. Metricel VM-1, Versapor 6424, Acropor AN5000, GF/A Glass fibre and GF/E Glass fibre — Gelman Instrument Co., Ann Arbor, Mich. Millipore SMWP 5.0 µm — Millipore Corp. Nucleopore — General Electric. Sartorius SM11301 — Sartorius Membrane Filter GmBH, Gottingen, West Germany.

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CHAPTER 5

DUST SAMPLERS

The major components of the sampler are: size selector, filter holder, filter, flow smoother, pump and motor, and battery. There are also various optional components such as flowmeter and running time indicator. This chapter discusses laboratory work carried out on dust samplers other than filters which were discussed in Chapter 4. Details are given in Appendices F and G.

5.1. Respirable Size Selection

It was found using simple comparisons in a laboratory dust cloud that many commercial dust samplers showed unexpected variation in indicated respirable dust concentrations attributed to the following effects:

1. pulsating airflows from single acting pumps leading to an increase in dust deposition in a cyclone size selector compared with smooth airflows;
2. passages between size selectors and filters modifying the size selection of the dust reaching the filter;
3. electrostatic effects under dry laboratory conditions;
4. variations in dimensions of machined cyclones.

These effects require that calibration of the size selection of complete dust samplers be carried out. The standard method using monodisperse dusts would have required a lot of time both to set up and operate (1). Thus, at Elliot Lake a simpler method was chosen comparing with a standard dust sampler in a dust cloud prepared in a specific manner. The horizontal elutriator built to the British Medical Research Council Specification (2,3) was chosen for a number of reasons — its availability, its expected 25% greater dust collection than the ACGIH specification, its simple theory, and the apparent variability of instruments intended to meet the ACGIH specification (4).

The choice of size selectors for the Elliot Lake program was reduced to the Casella 10-mm metal cyclone. However, this showed some variability traced to variations in dimensions, and was fairly heavy. Although the 10-mm nylon is attractive because of low cost and high reproducibility, its effective size is only about 70% of the Casella cyclone.

Eventually an impactor size selector-filter holder was designed which was smaller than either cyclone, and because the main critical dimension was one drilled hole, it was not subject to machining variations; also its sensitivity to pulsating flow was less than that of a cyclone. The first unit used a single-hole single-stage impactor, but after collecting about 2 mg of dust, part of the oversize could easily be loosened and penetrate to the filter. This was overcome by using a two-stage impactor, each stage having single holes. The unit is described in Appendix G.

The effective flow rate or size cut of the impactor can readily be changed by changing the hole size and it is thus a very flexible design. To date the sampling head has been produced in three versions:

1. normal flow, 2.0 L/min,
2. low flow, 1.5 L/min,
3. "open filter" in which the impact block is omitted, which however still leaves some, as yet unmeasured, size selection as with all "open filter" configurations.

It can be expected that any dust sampler fitted with a size selector can reasonably match specifications for lung deposition by adjusting the flow rate. Table 5.1 shows the expected equivalent flow rates at which some samplers would match the ACGIH and the BMRC specifications in a comminuted mineral dust cloud (2,3).

Cyclones and impactors show two important advantages compared with horizontal elutriators — freedom from orientation effects and, in mineral dusts at least, virtually no change in mass of "respirable" dust collected with a change

Table 5.1 - Size selector comparisons

	Size selection criterion	
	ACGIH	BRMC
Expected airflows L/min to match size selector		
Casella 113A horizontal elutriator	1.7	2.5
CAMPEDS	2.5	2.0
Casella cyclone		
10-mm nylon cyclone		
coal mine Cassette[1]	1.7	1.4
Millipore filter holder[1]	1.3	1.0
Expected relative respirable dust concentration		
Mineral dusts	1.0	1.25
Diesel exhaust particulates	1.0	1.0

[1] Tests indicated substantial differences between the two filter holders supplied with the M.S.A. sampling head, whether this applies to other makes or to more recent units is unknown.

in flow rate. Thus, flow rate control is not very important and the "respirable" dust concentration should be calculated using the nominal flow rate rather than the average during sampling.

5.2. Sampling Position

One study showed that on average a personal sampler indicates a 25% higher dust concentration than does a fixed position sampler. A fixed position sampler was hung as near to the miner as possible to avoid likelihood of damage. Stope drillers often moved more than 20 ft during the shift and although slusher operators spent most of their shift at the controls near the sampler, they also spent some time in the stope adjusting cables, etc. Fixed position long period sampling requires more effort to obtain proper samples than simply issuing personal samplers on surface.

Thus, for a routine program personal sampling is probably simpler and gives a better indication of exposure. Fixed position sampling and observation may be required to identify dust sources and determine corrective measures.

In personal sampling, the sampler intake should ideally be close to the nose, but this is a nuisance to the wearer and a position on the lapel is the usual alternative. For miners, a pump and sampling head attached to the cap lamp battery would be the least hindrance and least subject to damage. A few tests were carried out to examine the effect of position of the sampler on indicated dust concentration.

In these tests each miner wore two Casella personal samplers with one sampling head on his lapel and a second on his belt. The samplers were worn throughout the shift as described previously (5). The results expressed as a ratio of respirable silica dust concentration measured at the belt to that measured closer to the breathing zone at the lapel, are shown in Table 5.2.

The mean ratio of dust concentration at the two positions — excluding readings with insufficient samples and those with sampling heads covered by oilskins — is 1.04 with a range of 0.7 to 1.6 suggesting that the belt position is

Table 5.2 - Comparison of lapel and belt sampling positions

Operation	Ratio of belt to lapel dust concentrations					
Drilling	0.7,	1.1, 1.0,[1]	1.0,[1] 0.5,[2]	2.0,[1] 0.25[2]	1.2,	0.8,[1]
Slushing		1.0,	1.1,	0.8,	1.2	
Haulage	0.7,	0.2[1]				
Muck and clean up	1.0,	1.6				

[1] Ratio inaccurate as sample weight was small.

[2] Sampling head on belt covered by protective clothing (oilers).

satisfactory unless protective clothing is worn over the sampling head.

These results suggest that combining the sampling head with the caplamp battery could be the most satisfactory compromise between accuracy of sampling and wearer comfort for general mining operations. However, it leaves two problems:

1. Possible interference from clothing which requires training and checking by supervisors to assure that the battery-mounted dust sampler is worn outside all clothing.
2. The possible pronounced over- or under-estimation of dust levels if the source is above or below the operator such as on a sorting table or at screens feeding a crusher.

Another alternative, the Casella SIMPEDS has the pump on the battery and sampling head on the cap lamp (6), but was rejected because miners found the extra stiff connector to the helmet a nuisance and the dust deposit on the filter could be jarred and shaken off rendering the quartz analysis subject to error.

5.3. Filter Holders

The functions of a holder are to support the filter in a leak-free container and to present a uniform flow to the face. It consists of an inlet cone, a clamping device, an air seal, a backing plate, and an outlet connection. It has been found desirable to:

1. use mainly metal components, as erratic effects have been experienced with plastic inlet cones;
2. use an inlet cone with a minimum surface area, an included angle of 60°, and rounded corners;
3. let inlet and outlet masked areas of the filter coincide to give a uniform deposition area for X-ray analysis;
4. avoid rotation of the filter and consequent damage during clamping by using low friction washers of Teflon or nylon between filter and rotating member if any;
5. prevent leakage around the edge of filter into the outlet connection from both the inlet cone and the ambient air by using air seals;
6. prevent the silver membranes from sticking to the inlet cone which happens under some conditions, as yet unexplained. This is now avoided by using a thin plastic washer between the filter and the cone. The problem seems to occur most often in humid conditions and has occurred with aluminum, stainless steel and brass.

5.4. Flow Smoothers and Flow Measurement

Most small battery-powered samplers have single acting diaphragm pumps with an oscillating suction. A continuous smooth airflow is preferred as it avoids modifying the size selection characteristics and gives a true reading on the commonly used taper tube flowmeters. Although both these effects could be compensated by calibration, there is no guarantee that the compensation would be uniform over the entire operating range.

The Elliot Lake Laboratory did some work in developing smoothers as described in Appendix G.

The smoothers examined consisted of one or more chambers in the suction line to the pump, each with a rubber diaphragm separating it from the ambient air. The major requirements identified were:

1. the connection between the final smoother and the pump should be as short and large as possible;
2. the volume in each chamber should be as large as possible;
3. the rubber diaphragm should be as thin or elastic as possible without collapsing under maximum suction — coil spring supports may help; and
4. there should be a restrictive orifice between each pair of chambers.

In measuring flow of dust samplers, errors due to pulsating flows have to be avoided. Taper tube flowmeters, because they read the root mean square flow, are not suitable as primary standards although they are almost universally used for sampling pumps. Bubble burettes, wet test gas meters, or spirometers can be used as primary standards for calibrating taper tube flowmeters.

Some dust samplers have built-in flowmeters and some have external units. The Elliot Lake Laboratory used external flowmeters attached to the air inlet to the size selector. These formed a check on the entire samplers, and in many cases low readings led to discovery of leaks in filter holders and smoothers which would not be revealed in a changed reading on the internal flowmeter as mounted in many sampling pumps. A simple leakage check for many samplers is to see if blocking the inlet stalls the pump.

Checks were made with the flowmeter underground. Very high readings were found in deep mines due to the response of the flowmeter to air density. This renders flow checking by untrained staff difficult and experience with the Casella pump is that only occasionally during a shift do significant changes in airflow due to high dust loading occur. One mine found overloading only on 15% of the samplers issued to operators of diesel powered Scooptrams. It is thus better to set up the pump for maximum stability rather than for easy flow adjustment, and to accept that thick samples may be slightly underestimated because of the drop in flow rate.

5.5. Sampling Pumps

CANMET's experience has been mostly with the Casella pump and has indicated the following features:

1. A by-pass flow adjustment is undesirable because it increases the drop in flow rate as dust thickness increases.
2. A throttle flow adjustment is undesirable because it limits the thickness of dust deposit.
3. The least response of flow to dust thickness was obtained using a stroke adjustment.
4. The response to pressure and temperature in a mine is complex and the flow characteristics of the pump and flowmeter should be calibrated over the possible pressure and temperature ranges.
5. A running time indicator is useful for indicating faulty samples due either to malfunction or being turned off by the wearer.

The Casella SIMPEDS pump used in the Elliot Lake Laboratory has adjustments for both motor speed and stroke and is fitted with a running time indicator.

The response to pressure of depth in the mine was investigated in the laboratory. It was found that the flowmeter reading increased steadily with depth while the true airflow at that pressure decreased. The response to temperature was small and was opposite on the two units tested. However, erratic changes of up to 5% in airflow have been noticed when the pump differed in temperature from the ambient air. The details are given in Appendix G.

5.6. References

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CHAPTER 6

DISCUSSION

In judging the suitability of the personal sampler for assessing pneumoconiosis hazard and for reporting the results, it has been necessary to develop standards for permissible dust levels. Perusal of published standards in the world and laboratory correlation between instruments suggest that the threshold limit values for mines should lie in the ranges of (1):

> from 2.0 to 4.3 mg/m^3 for total respirable dust, from 0.5 to 3.0 mg/m^3 for respirable combustible dust, from 0.06 to 0.5 mg/m^3 for respirable quartz dust.

Sensitivity of the method is about one tenth the lower limit of the above range for each component respectively.

So far this report has only considered the pneumoconiosis hazard of the airborne dust. However, a number of studies have suggested a relationship between bronchitis and dust exposure, and it seems possible that in future dust standards may be controlled by the bronchitis hazard as well as by the pneumoconiosis hazard. Although very little is known about parameters of dust affecting bronchitis, it presumably is that which is deposited in the bronchia and thus coarser than the respirable dust fraction considered for pneumoconiosis. A better terminology might be 'alveolar dust' for that entering the deep portion of the lung, avoiding the term 'respirable' as this implies that entering the nose. The dust collected in the second impact stage of the CAMPEDS cassette could form a useful estimate of dust in the bronchial tract and thus of the bronchitis hazard. Possibly a target value should eventually be set for a mass concentration measured thus.

6.1. Dust Sampling

Preliminary results in mining showed that personal dust sampling instruments indicated 25% higher dust concentrations on the average than samples fixed in position in the working place. It was also shown that dust concentrations vary greatly with time. For assessing dust exposure it is clear that long period personal dust samplers are most suitable.

The Canadian mining personal dust sampler was developed by the Elliot Lake Laboratory for convenience in use by underground miners and is called the CAMPEDS. A cassette is used to minimize filter handling outside the laboratory and the sampler is mounted on and is powered by the cap lamp battery. It has had ready acceptance by miners and by ventilation staff. The mines need experience in using it to develop a system for switching it on, issuing and returning the sampler, for recording the relevant data and in interpreting the results and applying them to dust control.

Because the cap lamp battery is heavy, a version using a much lighter battery has been built for use where a cap lamp is not required, and this can be used either as a personal sampler on surface or as a fixed-position sampler underground.

It has been shown that alterations in any component of a size selective dust sampler may affect the size selection characteristics. Calibration of the complete sampler as intended for use is clearly desirable.

6.2. Gravimetric Assessment of Dust

This assessment is susceptible to errors as described in Appendix H. It is found that 90% of the samples have errors less than 0.1 mg; some 3% of the samples have substantially greater errors than this due to loss of filter material in handling or to errors in transcribing.

It should be appreciated that these errors in weight of dust can result in very large errors in calculating percentages of quartz in airborne dust. Reasonably accurate percentages can only be obtained with thick samples or by calculating a mean from a number of samples. Thus the quartz quantity should be compared with the basic value rather than with the percentage formula for the threshold limit value for quartz (2,3).

6.3. X-ray Analysis

Anderson has reviewed the literature on free silica analysis and has shown there are a number of problems (4):

1. particle size distribution in the sample,
2. the amorphous high solubility layer on the surface of quartz particles,
3. the various forms in which free silica occurs in nature, and
4. interference.

In addition to these problems there are related difficulties in interpretation of the analysis for estimating the health hazard, and these are:

5. biological effects of composition and structure, and
6. synergistic and inhibitory effects.

In mines, free silica occurs mainly in the coarse crystalline form of α-quartz. There are also some occurrences of the fine crystalline varieties such as chert. Crystobalite and tridymite can occur in younger rocks. Most samples analyzed in the laboratory have contained the coarse crystalline form of a quartz.

Particle size distribution of quartz from comminution of quartz-bearing rock tends to be constant below the original grain size, because it is usually the strongest mineral present. Because the original grain size is usually larger than the respirable size, it is found that the quartz particle size distribution in the respirable portion of the airborne dust collected near the dust source is reasonably constant (5). Some loss of the coarser respirable particles occurs by settlement, etc. when samples are collected at a distance from the dust source. However, the largest proportion by mass of the quartz particles falls into the most sensitive size range for XRD analysis, and this effect is unlikely to lead to significant error using XRD.

The amorphous high-solubility layer on quartz particles appears to be an effect of heat and stress in crushing, but is not likely to cause significant error in quartz analysis by XRD of respirable dust samples from comminution, as most of the quartz would be above the 1 μm size, at which a significant effect occurs (4).

Both these effects would in part be compensated by calibrating with a quartz material produced by a comminution process.

The various forms in which free silica occurs in nature certainly raise considerable difficulty for the analyst, a specific study of the X-ray analysis of varieties of crystalline silica is given in Appendix E. This suggests that a coarse crystalline form of quartz should be used as the main standard in mining. The errors in using this for fine crystalline quartzes may rise to 40% but this is probably insignificant in relation to the unknown differences in toxicity and their rare occurrence. The errors in analysis could be considered as part of their structure to be examined in epidemiological studies with their concentrations expressed as XRD equivalent in coarse crystalline quartz. Calcined diatomaceous earths have been analyzed for crystobalite with success, but tests on tridymite showed only low intensity lines.

Interference has been found to be very common, but is usually detected and circumvented by making measurements on more than one diffraction line and is minimized by using as long a wavelength as possible.

The effects of composition, structure, synergism and inhibition require elucidation from both laboratory experiments and epidemiology. However, epidemiology will be of little value without good estimates of exposures to quartz and other dusts in the field which can in the meantime be measured in terms of an analytical standard based on a specific quartz.

In addition to the above factors, the XRD analysis of quartz is limited in accuracy and sensitivity by the following:

1. lack of accuracy of size selection and airborne dust sampling procedures,
2. gain or loss of dust between time of collection and measurement,
3. evenness of dust deposit and presentation of a representative portion to the X-ray beam,

4. penetration of dust into filter and hiding from X-ray beam,
5. absorption of X-rays by the dust layer,
6. stability of X-ray intensity and equipment alignment,
7. variations in background XRD from a clean filter, i.e., small, apparently random, peaks,
8. errors in data handling.

Size selection and sampling procedures are common to all respirable dust analyses and calibration on complete systems is desirable if not essential. The dust samples as collected on filter membranes are easily damaged and have to be handled with care.

Uneven distribution of dust over the surface of the filter occurs on most types of sampler, usually with the centre being thicker. Systematic differences between types of dust samplers have given 40% differences in the ratio of XRD intensity to mass of quartz and the effect on analysis is minimized by calibrating with samples collected by the selected sampler in a pure quartz dust cloud.

Fine dust penetrates into the pores of the filters used at the start of sampling, before a dust bed builds up. This dust may be hidden from the X-ray beam by the filter material and the effect is allowed for by using thin sample correction factors determined during calibration with samples collected in a pure quartz dust cloud.

Absorption has only been found to be a problem on heavily laden filters. All 25-mm diameter filters so far examined which have less than 2 mg of total respirable dust (the target value for a full shift sample), as well as many heavier ones, have not needed significant corrections for absorption. However, substantial absorption has occurred on 13-mm diameter filters, and samples thin enough to avoid absorption corrections are too light to weigh accurately.

The stability of the X-ray intensity is dependent on the high voltage generator, X-ray tube and alignment of the equipment. No short-term stability problems have been detected but a regular decrease of about 20% per 1000 hours of operation does occur. The main cause of this is believed to be pitting of the target by the electron beam generating the X-rays. The long-term drift is controlled by the use of standard permanent samples prepared from a quartz and setting resin mixture.

The small background XRD peaks from the clean silver membranes limit the sensitivity to between 0.5 and 10 μg for the various diffraction lines of quartz. This limit has been found to correspond to a maximum counting time of 100 to 200 seconds per step. A counting time of 10 to 40 seconds is normally used.

Errors in transmitting data over the 350-mile phone line to the shared-time computer are frequent, leading to detectable errors in about 1 in 3 samples and gross errors in about 1 in 10 samples. During thunderstorms the error rate is higher and the computer is unusable. Enough redundant items of information have to be returned from the computer to detect significant errors; probably many, leading to errors in quartz estimates of less than 20%, slip through. Data transcription errors occasionally occur in the production of punched paper tape.

Repeat quartz analyses four weeks later have shown differences of 10 μg or 3%, whichever is the greater (Appendix C). Comparisons (6) between analyses by this and other laboratories on sets of field samples have shown that some are in good agreement with CANMET results, but others range from half to one and a half times as much quartz. It was suggested that these differences arose largely from the calibration procedures not being matched to the specific field samples examined.

6.4. Sampling and Analysis Time Study

The CAMPEDS is intended to take full shift samples in routine analysis and for this about 1/4 hour per sampler per day is required to issue, receive, change filter cassette, check sampler, and keep records. The CAMPEDS has not been in use long enough to know maintenance requirements, but based on experience with the earlier Casella pump, the loss of samples due to various causes is expected to be less than 10%, that samplers would require repair or maintenance about once per month, and that one full day per month should be sufficient to maintain and repair 5 samplers.

Weighing is done with the Elliot Lake Laboratory Mettler Grammatic balance and has taken about 2 minutes for both tare and gross weights per filter; ashing requires about 5 to 10 minutes per sample in batches of 6, mainly because the oven does not have thermostatic control and ashing temperature is fairly critical — too low and the filters do not ash fully, too high and the silver membrane filters distort.

A time study of the X-ray analysis procedure is given in Table 6.1 which shows the time required for each operation and how it could be fitted into the working day. It is based on a one-batch unit, i.e., an automatic sample changer magazine containing 2 standards and 33 samples.

Throughput can be compared with the most likely Canadian requirement which is estimated at 20,000-30,000 samples per year based on a total of about 10,000 miners working with medium- to high-quartz ores and samples from about 10% of the remaining 40,000 Canadian miners in low-quartz mines at two samples per year.

6.5. Composition of Airborne Dust

To date over 5000 airborne dust samples have been analyzed for quartz and over 2000 for combustible dust and their results are summarized in Table 6.2. The analysis of mine working place samples and special dust studies measuring air entering and leaving the working place has led to a fairly clear understanding of the main factors affecting the composition of dust in mine air as follows:

1. The quartz content as measured by X-ray diffraction and expressed in terms of the ash portion of the respirable dust is usually less than the quartz content of the ore and agrees reasonably well with that measured in the fine-sized fractions of ore

Table 6.1 - Time study of analysis procedures

Batches per day	1	2	3	4
Magazine change time[1]	9	9,14	9,14,20	7,12,17,22
	Technician time required, h			
Sample handling, setting X-ray	1/2	1	1 1/2	2
Computer operation	2	3 1/2	5 1/2	7
Calculating, checking, reporting	1	2	3	4
Totals	3 1/2	6 1/2	10	13
Samples/5-day week[2]	100	180	280	360
Technicians required	1/2	1	1 1/2	2

[1] Hours on 24-hour clock.

[2] Allowing for repeating heavy samples on a second line and only running four nights/wk.

Table 6.2 - Composition of the respirable airborne dust in various mines: ranges and means

Mine		Combustible dust %	Ash %	Quartz as % of Total dust	Quartz as % of Ash	Quartz in ore %
A	Range	--	--	0-100		99+
	Mean[1]	--	--	80		
	Mean[1]	--	--	50		
B	Range	--	--	0-100		65
	Mean	--	--	25		
C	Range	8-91	9-92	0-70	0-100	65
	Mean	57	43	22	52[2]	
D	Range	5-100	0-95	0-15	0-30	30
	Mean	49	51	7.5	15	
E	Range	19-96	6-81	0-2	0-30	10
	Mean	50	50	1.5	3	
F	Range			0-15		15
	Mean			1.2		
G	Range			0-12		10
	Mean			1.7		
H	Range	10-95	5-90	0-10	0-20	40
		50	50	5	10[3]	

[1] Two means were obtained before and after the mean dust concentration dropped from 0.3 mg/m^3 to 0.1 mg/m^3 during a concentrated effort on dust control.

[2] Chemical analysis of one "rafter" dust sample from near the crusher gave, after removal of +5 μm sizes by sedimentation, 49%.

[3] Chemical analysis of "rafter" dust samples from near the underground crusher gave 15% free silica.

samples by other methods. The ash portion of the respirable dust in mines appears to be mainly of mineral origin. Presumably the main reason for the quartz content of the respirable sized mineral dust usually being less than that of the ore is that quartz is stronger than many other minerals and is therefore broken less readily.

2. The mineral portion of the airborne dust in mines has been found to be significantly

diluted by oil mist which is collected on filters and weighed as dust from lubrication of compressed air operated equipment and by particulates in diesel exhausts, as shown in Table 6.2. While not yet identified, atmospheric dust and solids in atomized water could form significant diluents. As an example, in two mines with ore containing 60 to 70% quartz, the quartz per cent in respirable dust ash averaged 52%, but in the total respirable dust in one mine it was 25% and in the other 10 to 15%. The first mine used a few diesel-engine powered locomotives for rail haulage, whereas the second used many diesel load-haul-dump units.

3. The sulphuric acid content of diesel exhaust particulates is about 50% by weight.
4. The major portion of mineral dust in mines is produced from ore handling and the control of dusty air leaving these operations should be very effective in reducing miners' dust exposure.
5. Quartz constitutes the main health hazard in most mines having more than 20% quartz in the ore. However, in some cases diesel operation gives rise to high concentrations of exhaust particulates. While there are no standards for these, it would be imprudent to wait for pneumoconiosis to develop before controlling them.
6. The average dust concentration measured in the limited number of mines examined with significant quartz in the ore are close to the suggested permissible level of 0.1 mg quartz per cubic metre.
7. Dust concentrations vary from day to day even though full shift samples are being taken. The ratio of maximum to minimum concentration was found to range from 1.5:1 to 30:1 in a group of 20 men examined for 3 to 6 shifts each.

6.6. Further Developments

Two main problems with the X-ray diffraction analysis of quartz which could possibly be improved are that data-feeding to the remote computer requires too much technician time both directly and in correcting for transmission errors, and that unevenness of the dust deposit leads to sampling errors.

The use of an on-line computer to control the X-ray equipment, to calculate the mass of quartz from the area, and to repeat measurements on second lines when necessary, would greatly decrease technician time by permitting one technician to handle at least 400 samples per week and would also reduce X-ray time.

The area of sample covered by the X-ray beam has already been increased by 20% by machining the slits controlling the "vertical" dimension, along the goniometer axis, and increasing the size of the beam. However, the full benefit has apparently still not been obtained because of limited response to the outer parts of the sample in the graphite monochromator. Changing the X-ray optics to permit the full diameter of the filter to be illuminated and modifying the detector system to respond to the outer regions of the filter could diminish problems due to uneven deposition. Possible approaches are a larger graphite monochromator or the use of a solid state X-ray detector. Further possible improvements are:

i) the use of a vacuum system with chromium radiation,
ii) the use of an X-ray target to give the same beam width as the receiving slit recommended could increase intensity or tube life by a factor of 2.

The dust sampling and assessment system as a unit has one major problem of making rapid dust assessments and permitting immediate remedial action to be taken or studies of control failures to be undertaken. This requirement can be met by weighing before and after ashing for mines with small proportions of quartz in the ore but is not possible with high proportions. Two possible approaches are either to use much cheaper equipment for measuring quartz or to use more sensitive, more accurate techniques for determining the mass of dust and ash on the filters.

Infrared absorption has been used for quartz analysis and the least expensive equipment costs about $5000, which would seem a reasonable expenditure for individual mines or mining camps. To set this up would require finding a technique and a filter compatible with the dust sampler (7), gravimetric assessment before and after ashing, and X-ray diffraction assessment of quartz. Interference can also be expected with infrared absorption and results would need checking by X-ray diffraction but the method could be of immediate use making corrections by XRD for long-term estimation of exposure.

A more sensitive assessment of total mass and ash could be based on β-ray absorption (8,9) and X-ray absorption (10). Low energy β-rays with very thin filters have been shown to give a sensitive measurement of mass with little dependence on atomic number. X-ray absorption depends on atomic number and both methods could probably be used and calibrated for each mine to give sensitive and sufficiently accurate assessments of total respirable dust and ash. In most cases these two estimates would probably also give respirable quartz dust with sufficient accuracy for immediate application.

6.7. References

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ACKNOWLEDGEMENTS

The author wishes to acknowledge the assistance of his many colleagues with the experimental work and discussions. The major contributors were: K. Gammie, W.M. Gray, G. Ireland, B. Kirk, R. Kowalchuk, T. Lee, J. Leroux, K. Lichti, H. Machacek, P. Sekera and W. Stefanich.

In particular the author wishes to thank B. Kirk and W. Stefanich, authors of internal reports MRP/MRL 75-78, MRP/MRL 77-57 and MRP/MRL 77-109, parts of which were included in this report.

APPENDIX A

DEVELOPMENT OF X-RAY INSTRUMENTAL TECHNIQUES

This appendix is devoted to a detailed description of the settings and modifications for each component of the X-ray equipment. It should be appreciated that the results given were obtained with one particular apparatus having some custom features and with optimum settings for analyzing small quantities of quartz. Usually only one variable was studied at a time using preliminary best compromises on the others. These recommendations should be used as a guide for purchasing and setting up the equipment.

The equipment, mostly manufactured in Holland and supplied by Philips Electronics Ltd. will be examined in a logical order starting with filter mounting and finishing with data printing. Results for the four strongest diffraction lines of quartz will be given where the difference between them was significant. The requirement for more than one quartz line to minimize errors caused by interference was discussed in Chapter 3.

A.1. Filter Mounting

A Philips automatic sample changer is used which restricts filter mounting methods and prevents rotation of samples because of close spacing in the magazine. Two mounting methods have been tried, both ensuring that the centre of the filter coincides with the centre of the X-ray beam. The axial mounting surface on the goniometer had to be machined to clear part of the filter.

For the first method, a 22-mm diameter hole was drilled through the mounting plates supplied with the unit and a recess 0.25 mm deep and 25.5 mm in diameter was turned to receive the filters. The filters are held in the recess by petroleum jelly dabbed on the supporting surface in two or three places with a brush and smoothed out with a mating cylindrical tool. The filter is placed in the recess and the edges are pressed down with a clean mating tubular tool. This method was used for most studies and worked well except in a few instances when distorted filters loosened from the supporting surface and became caught in the sample changer. Filters can be ashed after X-ray analysis as the petroleum jelly is burnt off.

In the second method, the recess was taken nearly through to the top surface of the mounts. The top surface was chamfered to permit full illumination of the surface of the dust deposit down to 20°2θ, i.e., the lowest angle for a quartz diffraction line using CuKα radiation. The filter is held in place by a spring clip one size smaller than the 25-mm diameter hole as a high retaining force is not required. The surface of the dust deposit is below the axis of the goniometer causing an error of about -0.25°2θ in the peak position which has to be allowed for in mineral identification. This method is now in routine use and has fewer problems than the first. The arrangement is shown in Fig. A.1.

For holding 13-mm filters a similar arrangement is used in circular plates 25 mm in diameter which fits into the 25-mm filter holders. For filters with larger diameters of 32 and 37 mm, similar mounts have been made which have to be changed manually. However, for most samples the 25-mm diameter centre is punched out and mounted in the 25-mm filter holders with automatic sample changing.

A.2. Generator and Ancillary Equipment

The ultra-stable generator, PW1140, has required a few minor repairs and the addition of switch guards to prevent accidental operation when working on the X-ray table. X-ray tube cooling has used recirculation from a tank fitted with a water-to-water heat exchanger because dirt in the mains water supply rapidly clogged the recommended filter.

A.3. X-ray Tube

The use of three target materials – molybdenum, copper and iron were originally examined systematically. This suggested the examination of two further materials – cobalt and

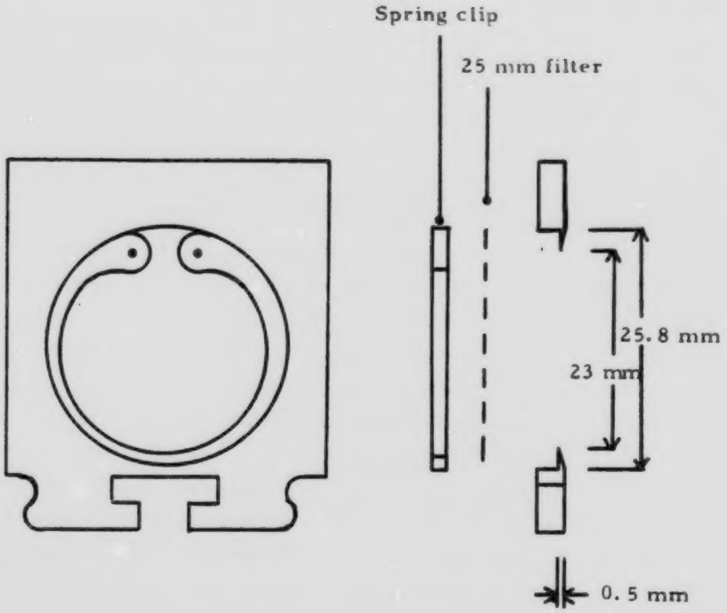

Fig. A.1 - Filter holder for automatic sample changer.

chromium. The following four quantities, which depend on target materials must be considered with respect to their effect on quartz analysis while maintaining other factors constant:

1. Intensity — area of diffraction line using the strongest emission line of the target, the unresolved $K\alpha_1\alpha_2$ doublet; high intensity shortens the analysis time.
2. Peak-to-background ratio; a higher ratio is associated with higher sensitivity.
3. Resolution, i.e., width of the line in equivalent "d" spacing. Clearly there is less chance of other mineral diffraction lines interfering with or overlapping the quartz line if the lines are narrower.
4. Wavelength of the characteristic X-ray affects absorption in the sample and therefore affects the limit of the linear range of analysis.

A.3.1. Intensity of characteristic radiation

Each tube was operated at its maximum rating as given by the manufacturer with the voltage and amperage adjusted to give the maximum peak height on the 1,0,1 quartz line. It was found that the optimum voltage based on intensity for the molybdenum tube was at or above the safe maximum voltage for the tube shield and it was operated at 55 kV and 18 mA. The copper tube was operated at 50 kV and 40 mA, the iron at 35 kV and 10 mA, and the chromium at 40 kV and 50 mA. The optimum for the cobalt tube was 40 kV and 50 mA.

Table A.1 shows the effect of target material on the intensities as peak heights of the diffraction lines from quartz and the silver membrane filter support. The first part of the table shows the values actually obtained and the second part shows the values corrected linearly for the energy input to the X-ray tube. It can be seen that the intensity for the quartz peaks increases from molybdenum through copper to iron even after allowing for the effect of "use" on the copper target. The chromium target gives slightly lower intensities than the copper tube did when new, possibly because of the effect of air absorption. The intensity of the peaks from the highly absorbing silver filter backing, however, falls as the atomic number of the target is reduced and the radiation is more readily absorbed.

The cobalt target was purchased after the copper and chromium targets were worn out and could not be compared directly. Records show that the cobalt target was not as intense when first installed, but improved after installation. These three targets give essentially the same intensity over the period of use considering the normal effect of wear.

Table A.1 - Intensities of diffraction lines (peak heights), relative to those using a copper target by net peak height

	Mo/Cu[1]	Fe/Cu[2]	Cr/Cu[2]
As measured			
Quartz 1, 0, 0	--	0.35	1.2
1, 0, 1	0.14	0.37	1.1
1, 1, 2	--	0.28	0.75
2, 1, 1	--	0.28	0.88
Average	0.14	0.32	0.98
Silver 1, 1, 1	0.85	0.2	0.31
Corrected for power[3]			
Quartz 1, 0, 0	--	2.0	1.2
1, 0, 1	0.28	2.1	1.1
1, 1, 2	--	1.6	0.75
2, 1, 1	--	1.6	0.88
Average	0.28	1.82	0.98
Silver 1, 1, 1	1.7	1.14	0.31

[1] When molybdenum radiation was used at the normal settings for copper radiation the resolution was poor and only the 1, 0, 1 line of quartz was measurable.

[2] These comparisons are unfavourable to a copper target because the copper tube had been operated for more than 1000 hr and its output was down to about 2/3 of the value when new, whereas the molybdenum, iron and chromium tubes were almost new.

[3] The tubes and settings used were:

	Focus, mm	mA	kV	W
Mo	1.0 x 0.1	18	55	1000
Cu	1.2 x 0.12	40	50	2000
Fe	1.0 x 0.1	10	35	350
Cr	1.2 x 0.12	50	40	2000

It is not clear what factors affect the decrease in intensity with time, but on the particular tubes used the life to half intensity at full rating was about 4000 hours on the copper and 3000 hours on the chromium. The cobalt tube was run at half rating and so far has run 4000 hours with only about a 20% drop in intensity, most of which has occurred in the 1500 hours since the rating was increased to 60%.

A.3.2. Peak-to-background ratio

Table A.2 shows the average effect of target material on the peak-to-background ratio obtained on a small number of field samples. It can be seen that the ratio increases as one progresses from molybdenum through to iron. Chromium was compared with copper on different samples at a later date and although preferable to copper, it apparently showed lower ratios than iron.

A.3.3. Resolution

Table A.3 shows the average effect of target material on the width of the peak as the mean on a small number of pure quartz samples. The width of the peak was measured at one-half of the net peak height above background, and was

Table A.2 - Net peak height to background ratio relative to that with copper target

	Mo/Cu[1]	Fe/Cu	Cr/Cu[2]
Quartz 1,0,0		1.5	
1,0,1	0.09	1.9	1.4
1,1,2		2.1	1.6
2,1,1		2.35	
Silver 1,1,1	0.17	1.37	

[1] When molybdenum radiation was used on normal settings for copper radiation the resolution was poor and only the 1,0,1 line of quartz was measurable.

[2] Comparisons were obtained at a later date on different field samples which had been analyzed earlier with Cu radiation on these two lines.

Table A.3 - Width of diffraction lines

		Absolute copper radiation	Relation to copper		
			Mo/Cu	Fe/Cu	Cr/Cu
By angle at 1/2 height in °2θ					
Quartz	1,0,0	0.25	1.6	1.0	1.0
	1,0,1	0.27	1.6	0.9	1.0
	1,1,2	0.31		0.9	1.0
	2,1,1	0.33		1.0	1.2
Silver	1,1,1	0.27	1.5	0.85	1.0
Average		0.29	1.55	0.93	1.0
By "d" spacing at 1/2 height in Å					
Quartz	1,0,0	0.05		0.8	0.67
	1,0,1	0.033	3.3	0.67	0.67
	1,1,2	0.0105		0.67	0.67
	2,1,1	0.0076		0.74	0.65
Silver	1,1,1	0.016	2.7	0.63	0.65
Average			3.0	0.70	0.66

expressed as an angle in degrees 2θ, or as equivalent increment in "d" spacing in angstrom units. The angular width is about the same for each line using one target with a marked difference between Mo and the others. The width in equivalent increment in "d" spacing decreases markedly with increasing angle and with target material in the order — Mo, Cu, Fe and Cr — suggesting that the longer the X-ray wavelength the greater is the resolution and the less likely is interference from minerals with close "d" spacings.

A.3.4. Absorption

Table A.4 shows the relative effects of target materials on absorbance by four samples. The three filters with samples had a lower silver peak than the blank with each target material being much more marked with the Cu, Fe and Cr radiations. This may be connected with tarnishing of the silver as the blank was a recent filter, whereas the others were 1 to 4 years old. There appears to be no significant differences in absorption between the three samples with any of the four radiations, suggesting that any of the radiations are suitable. However, studies on many samples are required to determine the upper limit of linearity for quartz analysis and to determine methods of detecting absorption as reported in Section 3.4.

The choice of target material depends on conflicting requirements and is a balance between higher absorption and improved intensity, peak-to-background ratio and resolution as the wavelength is increased.

It is clear that the disadvantage of low resolution and intensity when using a molybdenum target is not counterbalanced by its low absorption. The differences between the other four targets is not large and a high power cobalt tube is preferred because of the relative rarity of elements with high absorption for CoKα X-rays.

A.4. Take-off Angle

As the effect of increasing take-off angle in increasing the width of the diffraction line is much less than that due to other factors, efforts were made to maximize intensity. It was found that the advantages expected were not obtained at angles above 7°, presumably because of mechanical restrictions in the tube or tube shield and therefore 6° take-off angle given by the alignment tools is used. A specially designed or modified tube to give the same effective beam width as the receiving slit should provide greater intensities or longer tube life and be worth the possible extra cost. The Krottky tube will give a greater angle but some modifications to the tube shield and vertical goniometer are required.

Table A.4 - Absorption in sample - intensity of silver lines relative to those given by the absorbed radiation - Mo (Corrected for power) by net peak height

	Relative[1] intensity	Cu/Mo	Fe/Mo	Cr/Mo
Blank filter	1.0	0.9	1.02	0.27
Quartz 0.1 mg	0.85	0.57	0.58	0.16
Quartz 2.0 mg	0.88	0.64	0.66	0.24
Mine sample[2] 2.3 mg	0.8	0.54	0.54	0.15

[1] Relative intensity of the silver line from each filter using molybdenum radiation.

[2] The mine sample was chosen as containing some iron which has a high absorption for copper radiation.

A.5. Beam Height

The windows and shields on the goniometer were set to restrict the beam height along the goniometer axis to 10 mm. The later equipment by Philips Electronics Ltd. uses optics 12 mm wide. However, the new high-intensity tubes are supplied with a source 12 mm long and by machining all slits and shields to 12.5 mm, the illuminated area of the 22-mm diameter dust deposit was increased by nearly 20% and the intensity of the X-ray peak by about 15%.

A.6. Vertical Divergence of Incident Beam

The term "vertical divergence" is used in most texts to mean the angular divergence of the beam along the axis of the goniometer, even though with the goniometer used this is actually a horizontal direction. The vertical divergence is normally controlled to about 2° by Soller slits in the incident beam; that in the emergent beam is controlled by the graphite monochromator. If those in the incident beam are removed, its divergence is controlled at about 8° by source and sample. The effect for copper radiation of removing the Soller slits is shown in Table A.5 and Fig. A.2. There is a gain in intensity of 200 to 300% accompanied by a drop in peak-to-background ratio and distortion of the peak with a longer tail on the low angle side.

The removal of the Soller slits gives a considerable gain in intensity but at too great a loss in resolution. Coarser Soller slits might be a useful compromise.

A.7. Diverging Slit

The diverging slit regulates the width of sample illuminated by the X-ray beam at a set diffraction angle and tests have shown that intensity increases almost linearly with slit size until the sample is completely illuminated. The background also increases linearly and thus the peak-to-background ratio is constant until the beam is as wide as the sample. A secondary effect of increasing the slit size is to increase the peak width, but as this increase is less than 10% the gain in intensity far outweighs the loss in resolution.

Ideally the slit width should be set so that the beam just covers the sample at each of the angles used. However, the slits are manufactured in steps and the next larger slit width is used, with a slight loss in peak height-to-background ratio. Above 70°2θ it is not possible to illuminate the entire width of the sample and measurements using chromium radiation on the 1,1,2 and 2,1,1 quartz lines were carried out with less than the full width illuminated.

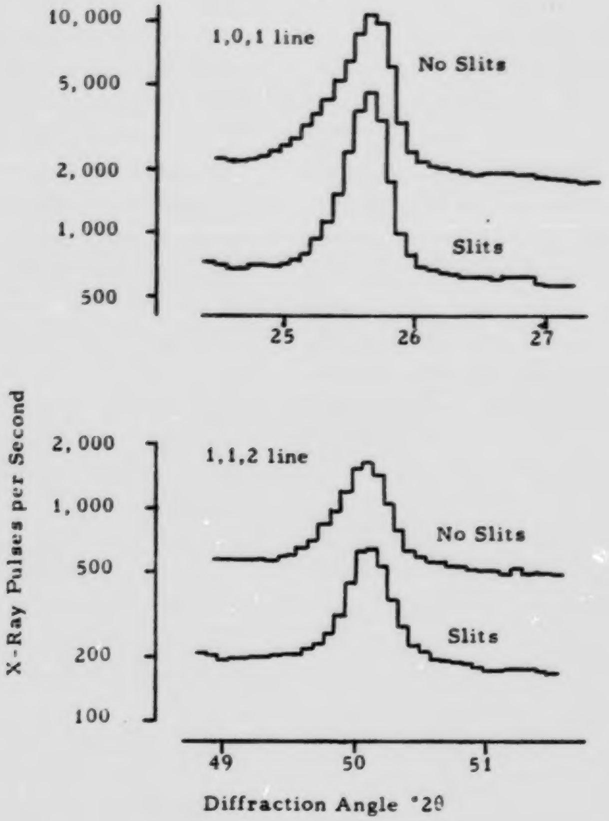

Fig. A.2 - Effect of Soller slits on diffraction lines from quartz.

Table A.5 - Effect of vertical divergence

	Ratio $\frac{\text{Soller slits in}}{\text{Soller slits out}}$ on quartz line			
	1,0,0	1,0,1	1,1,2	2,1,1
Intensity by area	0.38	0.40	0.37	0.39
Peak height-to-background ratio	1.45	1.45	1.2	1.05
Width at 1/2 height	0.83	0.85	0.82	0.85

Automatic adjustment of slit width to angle would be useful and a still further improvement for circular filters would be elliptical slits to match (1). To avoid variations in peak intensity due to slight misalignment of the samples, the width illuminated should be either greater or less than the sample in all its possible alignments.

A.8. Receiving Slit

When a crystal monochromator is used on the vertical goniometer, the receiving slit assembly differs from the usual arrangement in that the diffracted beam Soller slits are omitted and the scatter slit is mounted differently.

Table A.6 shows the effect of various receiving-slit widths on the peak area of the quartz diffraction line (1,1,2) using copper radiation and the diffracted beam monochromator. The gain in intensity more than compensates for the decreased signal-to-noise ratio as the slit width increases from 0.091 to 0.434 mm; however, it does not do so for the very wide slit at 1.5 mm. It is possible there is an optimum slit width at about 0.6 mm. The 0.434-mm slit width is about 10% of the width of the quartz diffraction line and does not degrade the resolution appreciably.

A scatter slit of about 4° angle based on the receiving slit as axis is used. Its removal, however, has not been found to increase background and the inner end of the beam tunnel has about the same angle of view.

A.9. Step Size

The system used counts the height across the diffraction line by step scanning. It has been shown that receiving slits up to 0.2°2θ can be used with a gain in intensity and there are advantages in both counting and computing time if only a few wider steps are used. Table A.7 shows the effect of step size and shifts in step position relative to the peak maximum on the integrated intensity using copper radiation of the 1,0,1 line of a pure quartz sample.

The corrected peak area is independent of step size in the range of 106 to 113 but shows some variation with the position of the peak within the step in the larger sizes. It is concluded that the variation found with the 0.083°2θ step size is within satisfactory limits, but that found with the 0.167°2θ is unsatisfactory. A step size in the range of 0.06 to 0.1°2θ is probably the most suitable and the values available depend on the drive. The goniometer drive is obtained from a motor controlled by a microswitch operated by a cam on the drive shaft with the variables — gear ratio and number of faces on the cam. The original motor supplied by the manufacturer ran on from 1/10 to 1/3 of a revolution depending on the friction applied to the shaft and was difficult to control. This was replaced by a motor fitted with a solenoid-operated clutch and brake which has a very small run-on and can be used with a multi-rise cam. However, the printout of the data for each step takes 4 to 5 seconds and control was modified to occur in the

Table A.6 - Effect of receiving slit width

Slit width		Peak	Intensity/unit width		Signal/noise
mm	°2θ	area	Total	Incremental	ratio*
0.091	0.03	28.8	317	317	3.7
0.203	0.067	62.8	310	304	3.5
0.253	0.084	74.9	296	242	3.28
0.434	0.144	102.8	227	154	3.02
1.5	0.5	147.2	98	42	2.58

*Expressed as (peak height - background)/background.

Table A.7 - Effect of step size and position relative to the peak maximum

Step size °2θ	Shift °2θ	Receiving slit width °2θ	Peak area Indicated	Peak area Corrected[1]	
0.021	-	0.03	1490	107	
0.042	-	0.03	737	106	
0.083	0.00	0.084	963	111	mean 108
0.083	0.04	0.084	929	106	
0.167	0.00	0.144	641	107	mean 113
0.167	0.04	0.144	716	119	
0.167	0.08	0.144	692	115	
0.167	0.12	0.144	665	111	

[1] Corrected for variations in peak area with slit width (Table A.6) and for number of steps (divide by number of steps per °2θ). This factor is effectively equal to change in counting time with step size.

same period. Using a 6-rise cam — the motor output shaft is 2 rpm — and the 1°2θ/minute gears, a step size of 0.083°2θ and motor advance time of 5 seconds can conveniently be obtained with minimum machining. The time constant of the one-shot circuit switching on the motor in the PW 1364 step scan control unit was reduced to 3 seconds by disconnecting one of the two capacitors.

A.10. Diffracted Beam Monochromator

The Norelco graphite monochromator replaces a filter to eliminate the Kβ radiation from the target.

A comparison between the graphite monochromator and the normal Ni filter on a pure quartz sample using CuKα radiation is shown in Table A.8. It can be seen using the monochromator that intensity is 40% higher and that the major gain is in the very great reduction in background of 2 to 6 times, increasing both with angle and with the presence of other materials mixed with the quartz. The peak width is essentially the same for both techniques.

The check on the resolution of the graphite monochromator was on the relative intensities of the Kα and Kβ peaks when the monochromator is set to maximum intensity for unresolved $K\alpha_1$ - $K\alpha_2$ or for Kβ radiation. The tests were carried out on a blank filter using the silver 1,1,1 peak and copper radiation. It was found that the intensity of the Kα peak was reduced by a factor of 1500 when the monochromator was set on Kβ rather than on Kα. The Kβ peak showed about the same reduction when the monochromator was set to Kα radiation. The Kβ peaks from the very strong lines of the silver metal filter do appear at intensities comparable with those of weak lines from mineral samples collected on the filter paper and should be recognized. However, they do not interfere in the analysis of quartz or crystobalite and "ghost Kβ" lines, as expected, have not been detected from any minerals as they very seldom reach 10% of the intensity of the silver lines.

A.11. Detector

The doped sodium iodide crystal and scintillation detector have been used throughout. Voltage applied to the detector has been 880 as this appeared to be in the middle of the plateau and the lower level and window width were set to cut off at 10% of full height above background. This reduces the total count by about 10% from that in the range-count minimum below the peak to the shoulder of the flat background above the peak. Output from the scintillation detector and monochromator is shown in Fig. A.3, both on the quartz peak and an adjacent background for copper radiation.

Table A.8 - Comparison of graphite monochromator and nickel filter using CuKα radiation

		Monochromator/Ni filter for quartz line 1,0,1	Monochromator/Ni filter for quartz line 1,1,2
Intensity:	peak height	1.35	1.55
	peak area	1.4	1.45
Peak height/background		1.9-2.7[1]	1.85-6.0[1]
Width at 1/2 height		1.05	1.0

[1] The gain in peak height-to-background ratio using the monochromator is greater for light samples and samples containing other minerals.

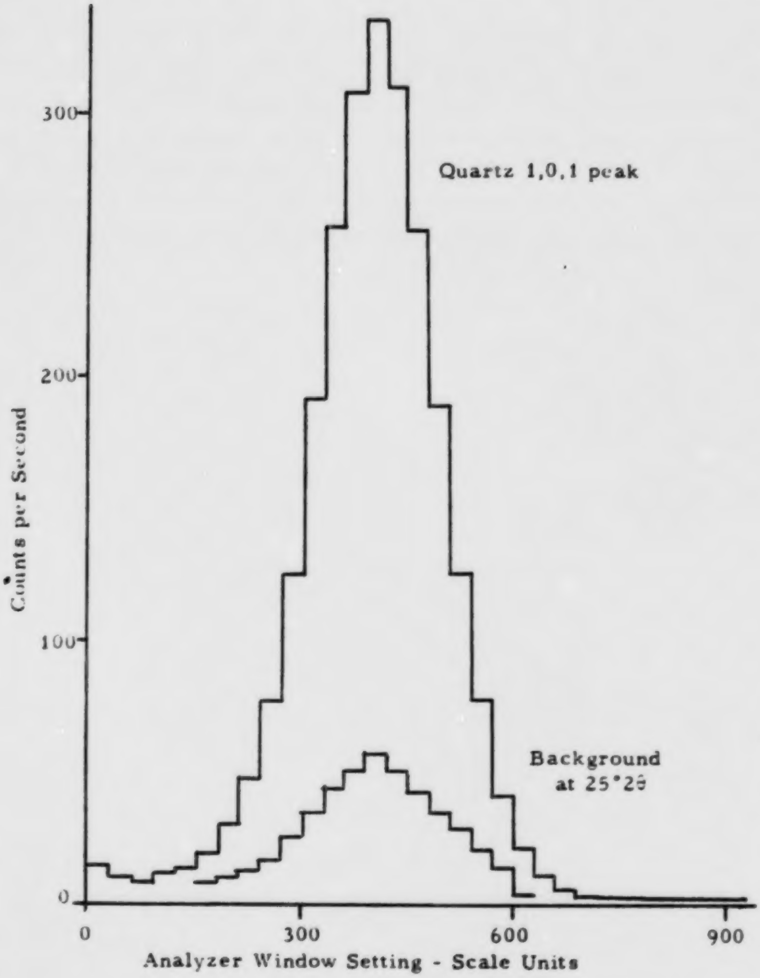

Fig. A.3 - Pulse size distribution from X-ray scintillation detector.

A.12 Evenness of X-ray Illumination Over Sample

Some preliminary tests were carried out with masks on the sample to check on alignment; however, the results were not conclusive, and a sample holder was made up in which a powder quartz test sample 3 mm in diameter could be set at any position in the sample area. The peak height from this test sample was measured on the 1,0,1 diffraction peak using CrKα radiation. The peak height was maximized by adjusting the diffraction angle, which varied over a range of 0.03°2θ and was approximately equal to the expected flat-sample correction. The background without the quartz sample was also measured and amounted to about 50% of the maximum peak height, much greater than the net peak height at the edges of the sample. The peak height was measured on grid points spaced 1.5 mm apart in both directions; Fig. A.4 shows the net peak height contours drawn in the sample plane. Also shown are the circles 10 and 22 mm in diameter outlining the two sample sizes used and the edges of the beam as defined by the shield components. It can be seen that intensity falls off very markedly towards the edges of the sample. As this fall-off could be due to variations in illumination or to variations in detector response, a photographic plate was placed in the sample position and exposed. This showed two deviations from uniform intensity — shadows of the plates in the Soller slit, and the expected drop in intensity with increased distance from the source.

The 3-mm diameter of the quartz test sample is larger than the Soller slit spacing and thus its effect should be smoothed out in Fig. A.4. The increase in distance from source to sample from one side of the sample to the other should be compensated for by the corresponding decrease in distance to the detector.

Thus it appears that the major cause of the decrease in peak height towards the edges of

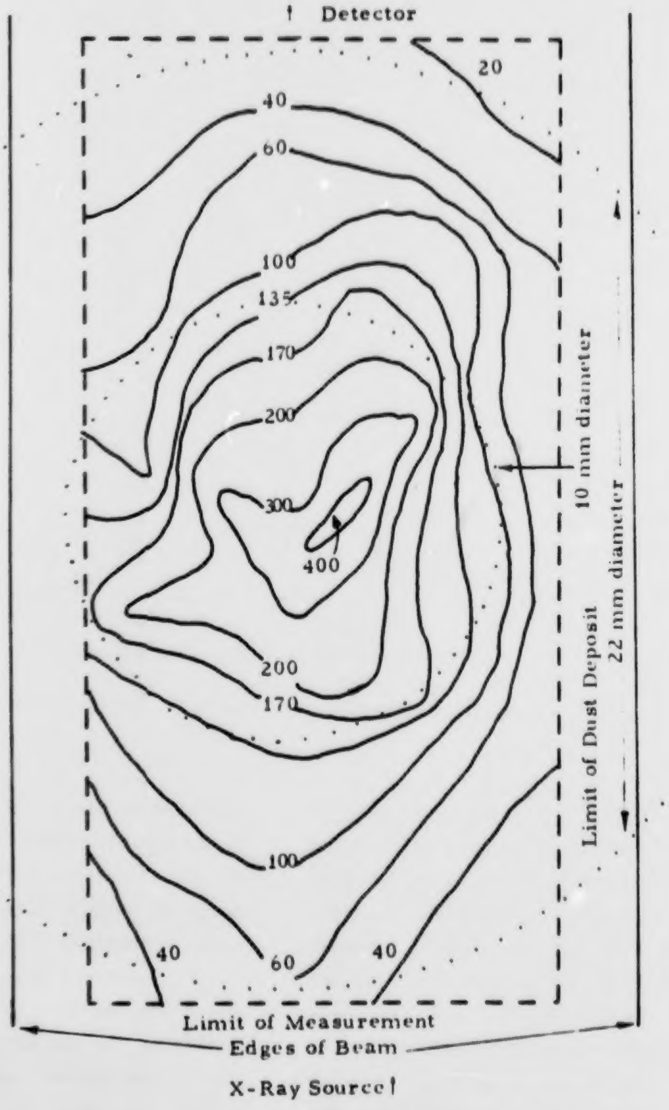

Fig. A.4 - Contours of diffracted beam intensity from a 3-mm diameter quartz sample — 1,0,1 line CrKα radiation.

the sample area is in the detection system. It is believed that the graphite monochromator crystal is the component responsible. It is possible that a larger crystal could give a greatly increased intensity from field samples. Philips Electronics Ltd. now supplies a larger crystal. Certainly an even response over the whole sample area would increase the intensity by a few hundred per cent. A possible alternative would be to use a solid state, lithium-drifted silicon detector in place of the monochromator and scintillator detector.

A.13. Noise Suppression

Spurious bursts of counts were observed on the chart recorder at irregular intervals when first received. Some of these were traced to the operation of electrical switches both in the system and external to it. The incidence of these bursts was greatly reduced by adding an extra stage of capacitor-resistance smoothing on the power input to the scintillation detector preamplifier. The manufacturers have since issued modifications which increase the smoothing of this power supply. A few other switches on the equipment also give noise problems and these must either be suppressed by fitting capacitors across the contacts or else not used during counting. The problems identified are:

a) step scan microswitch on goniometer drive — suppressed by adding capacitor across terminals;

b) switching recorder off is very noisy — suppressed by adding capacitors across PW 2500 supply terminals and ground;

c) fluorescent light on recorder table — removed.

Some spurious signals occasionally interfere with the control and data transfer signals producing difficulty in operating the computer program as well as unwanted stops in running through a set of samples on the X-ray equipment; their sources have not yet been identified.

A.14. Comparison of Angle and Energy Resolution Systems

Solid-state detectors for X-rays have been greatly improved, and the latest versions can resolve X-rays into fine energy bands. It has been suggested these detectors could be used for diffractometry instead of the normal practice of scanning through an angle with monochromatic X-rays (2). This would give the advantage that the whole spectrum would be received at one time; calculations suggested that the technique would be close to the technique now used in this laboratory.

An opportunity, by courtesy of Radionics Ltd., was found to test an Ortec high-resolution lithium-drifted silicon solid-state detector on the Philips X-ray equipment with a vertical goniometer, using a Northern Scientific multichannel analyzer.

A.14.1. Experimental technique

The energy resolution was compared with angle resolution diffractometry under as similar conditions as possible. The same diverging and receiving slits were used. The graphite crystal diffracted-beam monochromator (Philips part PW 1152) and scintillation detector were replaced by a scatter slit, Soller slits and the Ortec silicon solid-state detector, mounted on an elevating table.

Output from the solid-state detector and pre-amplifier, operated at liquid nitrogen temperatures, was amplified and counted on a Northern Scientific ADC and multichannel analyzer.

Three fixed diffraction angles were chosen based on the relationship between the energies of copper Kα (from the tube) and silver Kα (from the membrane filters used to collect the dust samples) radiations and the energies corresponding to the quartz and silver metal diffraction patterns.

Arrangement 1. The diffraction angle was set at 38.1°2θ where the CuKα coincides with the first line in the diffraction pattern of silver; the supporting filter is made from silver metal. The CuKβ almost coincides with the second line of silver. Fluorescence from iron and near elements can interfere with quartz.

Arrangement 2. The diffraction angle was set at 17.6°2θ where the AgKα coincides with the first silver diffraction line. This gives very little interference with the quartz 1,0,0; 1,0,1; 1,1,2; 2,0,2; and 2,1,0 lines. The arrangement might be improved by using a silver target tube.

Arrangement 3. The diffraction angle was set at 6.0°2θ where all the quartz diffraction lines occur at energies greater than the silver Kβ line.

A.14.2. Results

Some results from a pure quartz sample are shown in Table A.9. Arrangement 2 apparently gives the best results, and these are shown in Fig. A.5 to A.8. Figures A.5 to A.7 show comparisons between high and low quartz samples on both angle and energy resolution for the three main diffraction lines of quartz; Fig. A.8 shows results obtained by energy resolution for the three quartz lines 1,1,2; 2,0,2; and 2,1,0.

The results show that the diffraction peaks are broader for energy than for angle resolution and that this is likely to introduce more difficulties from interference by other minerals. This was observed on samples of mine dusts known to contain minerals interfering with some of the quartz lines. Some gain in resolution might be obtained by improving the arrangement but as the diffraction line is only 1-1/2 times as broad as a fluorescence line there is not much room for improvement without an improved detector resolution. The detector used had a width of 300 keV at half height for CuKα radiation.

The energy resolution technique gives much lower peak height counts than the angle resolution technique but as it gathers all its data simultaneously rather than consecutively, this disadvantage is partly offset. The difference between the two as tested is about 1700:1. The energy resolution detector is smaller than the X-ray beam and the energy technique could probably be improved by a better choice of

Table A.9 - Results from pure quartz sample

			Energy resolution			
	Angle resolution		Set up 1	Set up 2		Set up 3
	101	112	101	101	112	101
Diffraction angle °2θ	26.64	50.12	38.1	17.6		6.0
Diverging slit	1.0	2.0	1.0	0.5		0.25
Scatter slit °2θ	(1)		1.0	0.5		0.25
Receiving slit mm	0.56	0.156	0.156	0.156		0.156
Detector	scintillation		(2)			
Analysis	PHA		MCA			
Scanning	0.04°2θ steps		1024 channels, 0.05 keV/channel			
Channel width = d A	0.005	0.0014	0.03	0.011	0.0033	0.005
Width at 1/2 height = d A	0.025	0.009	0.22	0.085	0.03	0.11
Peak-background cps	2700	175	2.56	1.55	0.06	0.175
Area cps · d	68	1.6	0.56	0.132	0.0018	0.019
Peak-background	14.3	1.95	0.46	11.4	2.1	21

(1) Graphite monochromator is used; eliminates interference by fluorescence.

(2) Ortec 6-mm diam, 3-mm sensitive depth lithium drifted silicon.

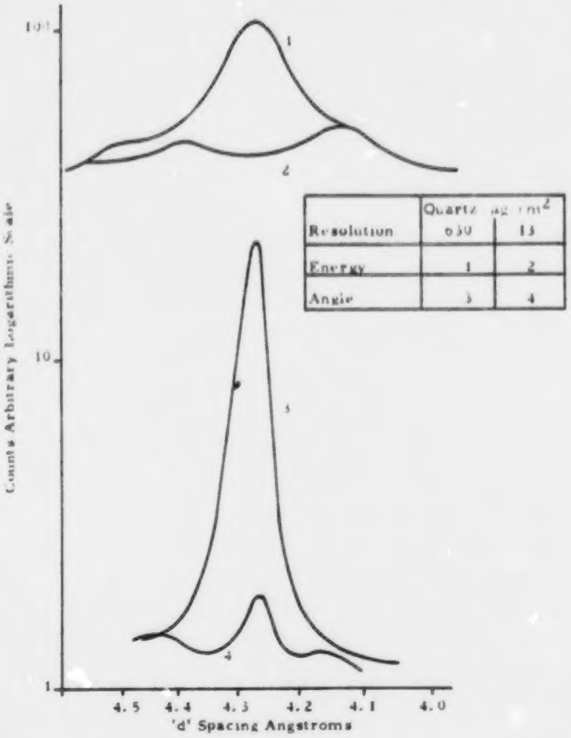

Fig. A.5 - Comparison of energy and angle resolution for samples with high and low densities of quartz — 1,0,0 diffraction line of quartz.

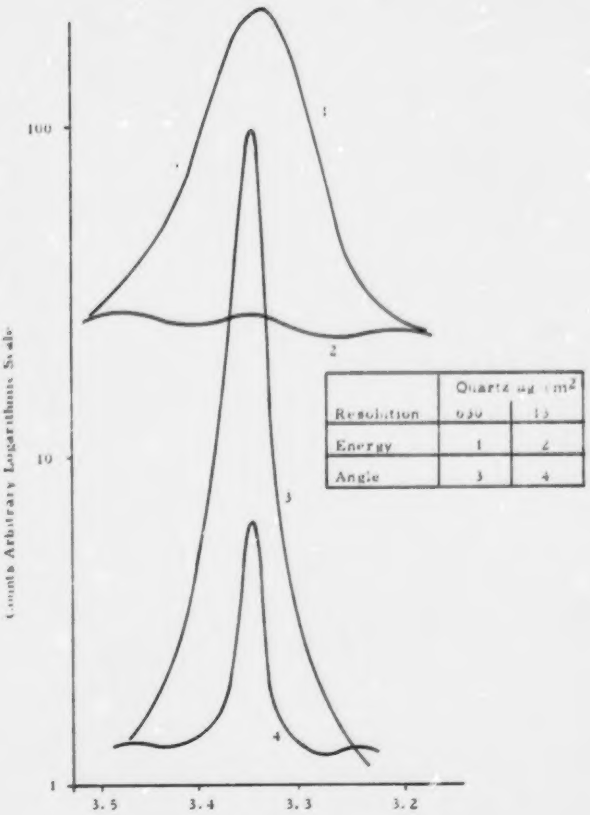

Fig. A.6 - Comparison of energy and angle resolution of samples of high and low densities of quartz — 1,0,1 diffraction line of quartz.

components making it comparable in sample time; however, there would be no advantage unless the resolution can be brought nearer to that of angle resolution.

The sensitivity for quantitative analysis of quartz using energy resolution was about one tenth of that using angle resolution. The lower sensitivity is due to the broader diffraction lines suffering from interference.

A.15. Data Printout

The data printout as originally supplied consisted of three seven-figure numbers separated by single blanks together with carriage return and line feed control signals for each count. The three numbers were the step number, the time, and the count. This format took 7 seconds for the printout on each step and was wasteful because only 15 of the 26 characters were used. Sample change could only be detected by carrying out logic computations on the step numbers. A second numerator was added to count samples and the format was changed so that one seven-figure number was made up of a two-figure sample number, a two-figure step number, and a three-figure preset count. The blanks were replaced by commas to simplify computer input processing. This reduced the number of characters per line to 18

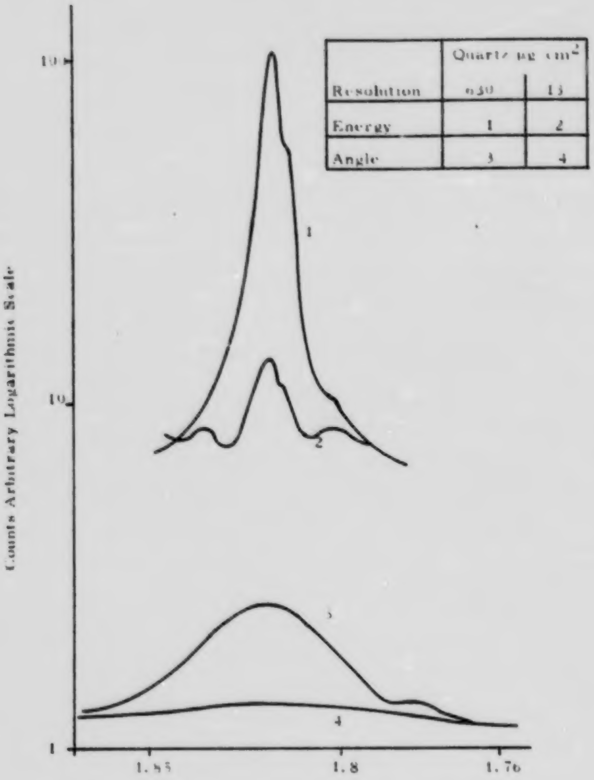

Fig. A.7 - Comparison of energy and angle resolution of samples with high densities of quartz — 1,1,2 diffraction line of quartz.

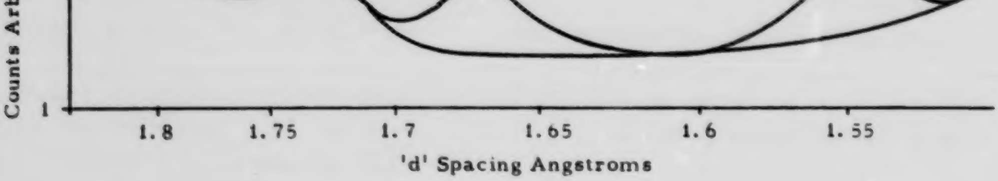

Fig. A.8 - Energy resolution chart for region covering the 1,1,2; 2,0,2 and 2,1,0 diffraction lines of quartz for samples with high and low quartz.

with a substantial saving in the time taken to transmit data to the computer.

A.16. References

1. Jenkins, R. and Paoline, F.R. "An automatic divergence slit for the powder diffractometer"; Norelco Rep; 21:1:9-14; 1974.

2. Giessen, B.C. and Gordon, G.E. "X-ray diffraction: new high speed technique based on X-ray spectrography"; Science; 159:3818:973; 1968.

APPENDIX B

CALIBRATION OF QUARTZ ANALYSIS

It was decided that the primary calibration of the X-ray analysis system would be carried out by collecting dust samples on filters from pure quartz dust clouds in conditions as closely as possible resembling field use. This would minimize any effect of particle size and systematic variations in particle deposition over the area of the filter and resultant errors in the proportion of quartz in the X-ray beam.

The first calibration was carried out on dust deposits collected on 25-mm diameter filters using Casella T13040 personal size-selecting gravimetric dust samplers (1).

B.1. Preparation of Pure Quartz Samples in Laboratory Dust Chamber

To calibrate the technique of determining respirable quartz in air down to the threshold limit value of 0.1 mg/m^3 of shift-period samples, it was necessary to prepare samples with as little as 0.05 mg of quartz. This quantity could not be weighed sufficiently accurately and the quantities of dust on the filters were determined indirectly.

A quartz dust cloud was prepared by continuous breakage and dispersion of silica sand (Ottawa, Illinois) into filtered air in the laboratory dust chamber using a twin-jet fluid energy mill. Samples were taken with four Casella personal samplers at 1.9-2.0 L/min and one Casella Hexhlett sampler at 50 L/min for various periods from 1 to 90 minutes (1). Filters from all the samplers in the longer runs were weighed and the ratios between the masses collected by the personal samplers and those by the Hexhlett were determined. Then, on the short runs, the masses of respirable dust collected by the Hexhlett were weighed and those on the personal samplers were calculated from these ratios.

The mass of quartz on the filters ranged from 0.04 to 3.3 mg, equivalent to a surface density of 10 to 850 μg quartz/cm^2.

B.2. Calibrations of Quartz Samples

Intensity measurements were made on the 101 quartz peak of samples collected by personal samplers in a pure quartz dust cloud using both copper and molybdenum Kα radiations. The results obtained are given in Table B.1 and are shown plotted in Fig. B.1 and B.2. Figure B.1 shows a plot of peak height against mass of quartz using a linear scale and Fig. B.2 shows the ratios of the peak height area to mass of quartz on the filter plotted against the mass of quartz. The logarithmic scale was chosen so that the shapes of the curves could be compared directly with one another.

The results show that the ratio of X-ray intensity to mass calculated from the mass of the 50-L/min Hexhlett samples of samples collected by the personal samplers decreases markedly for thin samples. This is presumably due to fine quartz either passing through or into the body of the 5-μm pore size silver membrane filters where it is screened from the X-rays. It is assumed that penetration decreases as the dust bed builds up and stops at 200 μg of quartz/cm^2. It is presumed that penetration is dependent on the mass of all dust collected and that a thin sample correction factor based on total respirable mass

Table B.1 - Calibrations of quartz samples (1,0,1 line) for copper and molybdenum Kα radiation

Mass of quartz μg/cm^2	Peak height C . sec^{-1}		Peak area C .°2θ . sec^{-1}		Signal/noise ratio	
	Cu	Mo	Cu	Mo	Cu	Mo
Samples collected by Casella personal sampler						
842	4700	774	1430	280	73	9.4
208	1198	194	357	66	28	3.4
121	690	110	210	37.4	17	2.3
99	545	83	156	26.4	14	1.6
52	244	36.5	72	12.6	6.5	0.8
33	137	21.4	47	7.0	4.3	0.5
22	88	14.6	29	4.6	2.3	0.45
10.5	41	6.6	13.4	2.0	1.1	0.17
10.5	39	6.3	13.6	2.1	1.2	0.16

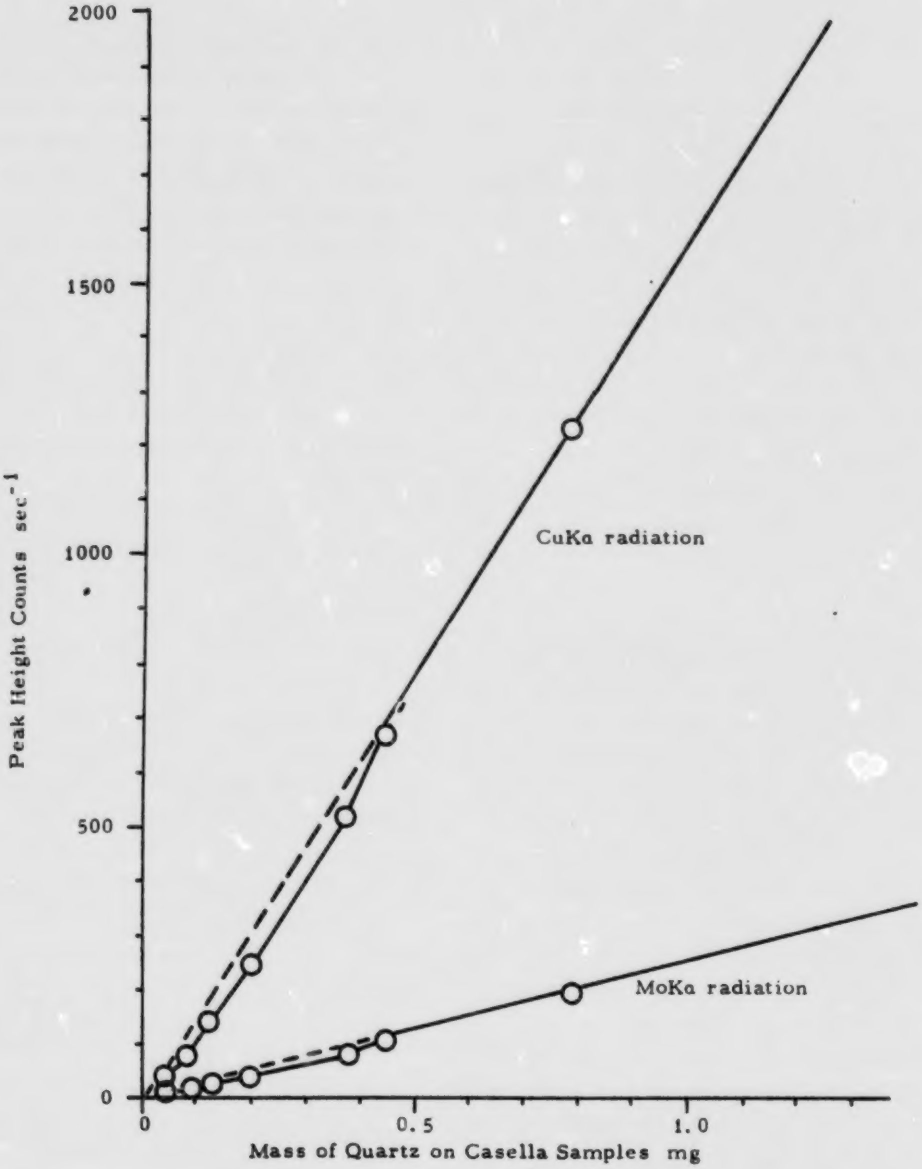

Fig. B.1 - Quartz calibration of X-ray with Casella samples.

can be applied. It can be expected this factor would depend on size distribution of the dust, penetration increasing with a finer size of quartz dust at the start of sampling but decreasing more rapidly as the bed of finer dust particles became less permeable. In practice, respirable-sized quartz shows little change in size distribution and total respirable dust tends to be finer in mines due to diesel particulates, and thus the correction factor would be overestimated, leading to a safe but small overestimate of quartz.

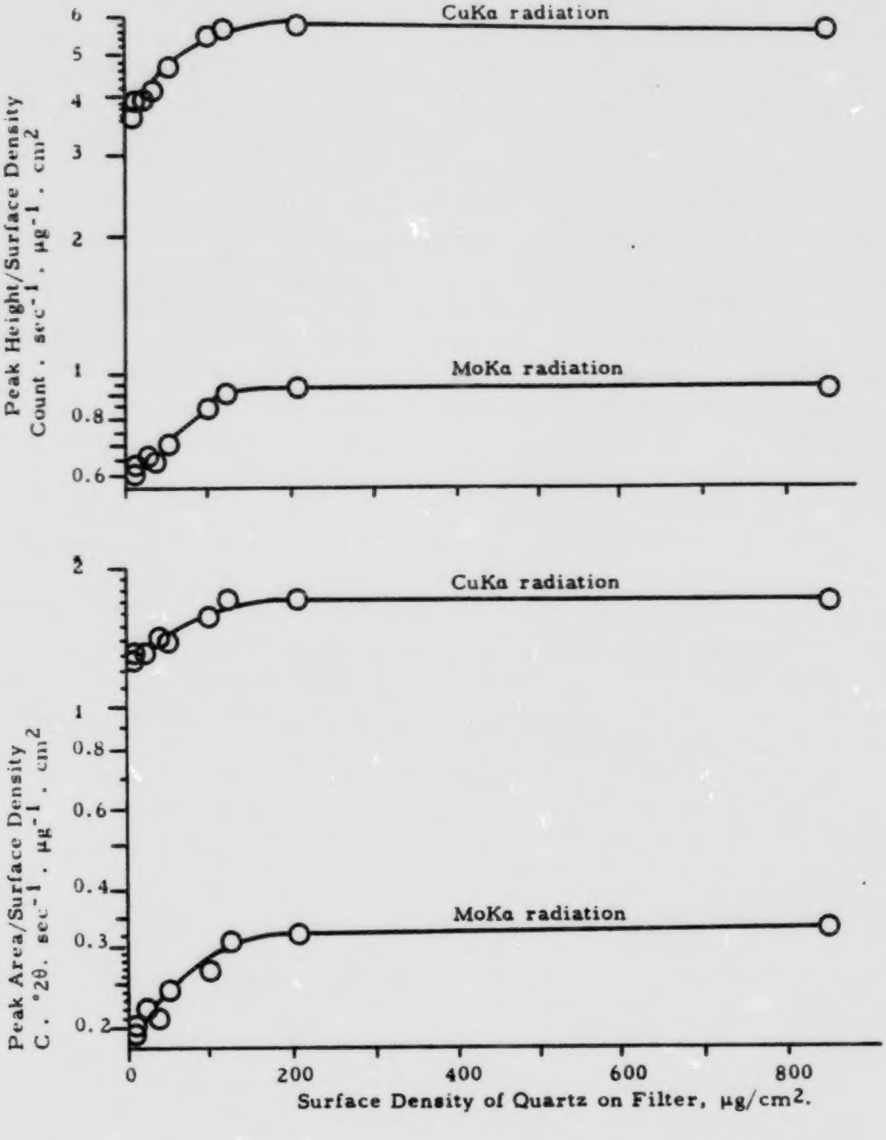

Fig. B.2 - Quartz calibration of X-ray with Casella samples.

B.3. Calibration of Other Dust Sampling Instruments Used in Elliot Lake Laboratory

A set of samples was collected in a pure quartz dust cloud using a number of dust samplers as listed below:

a) 2 Casella personal samplers, type 13040, airflow 2 L/min,

b) 2 modified Casella personal dust samplers with 37-mm diameter filter holders, airflow 2 L/min,

c) 1 open filter, 25 mm in diameter, airflow 1 L/min,

d) 1 open filter, 13 mm in diameter, airflow 1 L/min,

e) a set of 7 horizontal elutriator size-selecting samplers operated with either 25- or 13-mm diameter filters at a flow rate of 1 L/min.

Samples were also prepared from liquid suspensions which gave an even deposition of dust over the surface of the filter with the values given in Table B.2. It can be seen that the results are more variable than with airborne samples. In particular, the 1,0,1 line is stronger on the liquid dispersion than on airborne dust, suggesting that preferred orientation can occur.

The set of horizontal elutriator samplers was made with a range of airflow settlement area ratios to give measurements of particle size distribution in accordance with the technique proposed earlier (2). The X-ray intensity mass ratios are shown for the 1,0,1 quartz line on all instruments examined. Only measurements of reasonably dense samples have been included in this table to avoid the effect of lower intensity mass ratios on thin samples discussed previously.

Table B.3 shows there are appreciable differences in calibrating this range of dust samplers, most of which can be accounted for by

Table B.2 - Calibration of permanent quartz standards

Sampler description	Quartz line	Mass of quartz dust on standard expressed as equivalent on sample filter				
Filter size		22 mm diameter				13 mm
mm		St1	St3	St4	St5	St2
Casella personal sampler						
25	1,0,0	2.28[1]	3.55	7.4	5.56	--
	1,0,1	2.51	3.50	7.08	5.12	--
	1,1,2	2.51	3.63	8.01	5.78	--
37	1,0,1	5.9	8.23	16.6	12.0	--
Open filters						
25	1,0,1	2.87	4.01	8.1	5.85	--
13	1,0,1	1.52[2]	2.07[2]	4.4[2]	3.12[2]	0.765
HE[3](7.1 μm)						
25	1,0,1	2.89	4.03	8.15	5.90	--
HE(5.0 and 3.5 μm)						
25	1,0,1	3.04	4.22	8.53	6.17	--
HE(2.5 and 1.75 μm)						
13	1,0,1	--	--	--	--	0.72
HE(1.25 μm)						
13	1,0,1	--	--	--	--	0.93
25-mm liquid suspension	1,0,0	--	3.7	--	5.2	--
	1,0,1	--	2.6	--	3.9	--
	1,1,2	--	4.1	--	6.2	--
	2,1,1	--	4.7	--	7.4	--

[1] This standard shows a fairly high variability, ±10%, in repeat calibrations on the 1,0,0 line due apparently to the low peak-to-background ratio.

[2] This comparison was undertaken with a divergent slit large enough to cover the 22-mm dust deposit on the standard.

[3] Horizontal elutriator samplers.

Table B.3 - Calibration of dust sampling instruments

Sampler description			X-ray intensity[1]	
Filter size mm	Size selector Type	Size selector Size 50% Cut[3] µm	/Mass on filter	/Mass in beam[2]
37	Cyclone[4]	5	74	235
	Cyclone	5	68	216
25	Cyclone[5]	5	162	337
	Cyclone	5	172	358
	None	--	146	304
	HE[6]	7.1	145	302
	HE	5	139	290
	HE	4	138	287
	HE	3.5	139	290
	HE	1.25	74	154
13	None	--	232	249
	HE	7.1	247	265
	HE	5.0	255	274
	HE	3.5	256	275
	HE	2.5	245	264
	HE	1.75	245	264
	HE	1.25	190	204

1 X-ray intensity to mass ratios after correction for secular variation in X-ray intensity.

2 Calculated mass in the X-ray beam assuming even dust deposition over the active area of the filter.

3 Approximate aerodynamic size (diameter of unit density sphere with same falling speed) at which 50% of the particles are collected in the size selector and 50% pass through to the filter.

4 Modified Casella personal gravimetric sampler with 37-mm filter holder.

5 Casella personal gravimetric sampler.

6 Laboratory set of horizontal elutriator samplers.

two factors: uneven distribution of quartz over the surface of the filter — the Casella personal samplers show a visibly thicker deposit in the centre than at the edges, and the effect of quartz particle size which will be described in the next section.

B.4. Effect of Quartz Dust Size

The horizontal-elutriator size-selecting samplers remove some of the coarser dust particles before they collect on the filter. This set of samplers has a range of ratios of settlement area/airflow and thus rejects different fractions of the dust (2). The results are shown in Table B.4. The penultimate column shows the results of calculations for each increment of dust as the elutriators are steadily decreased in area/flow ratio and larger particles pass through and collect on the filter. The particle size range in which most of the increment of dust occurs is shown in the last column.

It can be seen that particle size has some effect on the intensity/mass ratio. The effect is not the same on both sizes of filter

Table B.4 - Effect of particle size on diffracted intensity of X-rays from quartz

50% size cut aerodynamic size μm	Mass on filter relative	X-ray intensity, arbitrary units	X-ray intensity/ Mass of qtz in beam		Particle size range of increments, μm
			Total	Each dust increment	
25-mm filters					
1.25	0.104	7.7	154		0.5-1.5
				308	1-4
3.5	0.42	54.5	270		
				334	3-6
5.0	0.60	83.5	290		
				342	4-8
7.1	0.78	113	302		
				322	6-10
Open	1.0	147	306		
13-mm filters					
1.25	0.104	20	207		0.5-1.5
				326	1-2
1.75	0.16	37	249		
				247	1.5-3
2.5	0.31	76	264		
				313	2-4
3.5	0.42	108	276		
				269	3-6
5.0	0.60	153	274		
				239	4-8
7.1	0.78	193	266		
				191	6-10
Open	1.0	232	249		

and is presumably confused with the effects of uneven deposition on the area and with different thicknesses. The results in Table B.4 suggest that the X-ray intensity/mass ratio is greatest for 3- to 8-μm aerodynamic size particles with a sharp decrease for particles smaller than 1 μm and a slow decrease for larger ones.

B.5. References

1. Casella Ltd.; London, U.K.; manufacturer.

2. Knight, G. "A simple method for determining size distribution of airborne dust by its settling velocity"; Amer Ind Hyg J, 33:8:526-532; Aug. 1972.

APPENDIX C

COMPUTER PROCESSING OF DATA

The data from step scanning over peaks is collected on punched paper tape and transmitted by telephone to a shared computer service in Toronto. Kemper and Van Kempen's program for γ-ray spectroscopy is the basis for determinating areas of the X-ray diffraction peaks (1). It was necessary to make minor modifications and prepare a program for reading data input and separating it into samples. In using this program it became apparent that some major modifications were desirable, as follows:

1. The original program had a set value of 2 for the "width". This was used for finding a peak by determining whether the count in channel N was significantly greater than counts in both channels N-2 and N+2. This could fail to find quartz peaks of low but significant intensity. The program was modified to make the width a variable input and a search was conducted for a peak which was the highest in a range rather than only being compared with a set channel on either size.
2. The program attempted to separate doublets such as the $K\alpha_1$ $K\alpha_2$ peaks of the high angle quartz lines into two areas and then obtained an unsatisfactorily high background between them. This was solved by using the variable "width" entry to indicate how far the program should scan over a secondary peak and decide whether it is separate or not. Values up to eight have been used for integrating close triplets (2°2θ) in some minerals.
3. The program originally computed the significance of a count relative to horizontal background. It was found that fewer errors occurred due to adjacent peaks if a linear sloping background was assumed. The first estimate of the slope is obtained from the first six and the last six counts in each sample. If the background slope found when integrating over the peak differs significantly from the first estimate, an error message is produced and the area can be redetermined with a new value for the slope.
4. The modified program also calculates the mass of substance in the sample from both the net peak height and the area by comparing with the values obtained on the standard. If the two values of mass are not close it is indicative of some form of interference or distortion in the filter. Distortion can be checked by visual inspection and should be noted when unloading the filters from the magazine.

The program used is in BASIC language and is given on the next few pages. The program has provisions for correcting input data on-line when alphabetic or non-type characters are introduced as quite frequently happens in data transmission to the computer in Toronto. Some redundant information is returned to Elliot Lake to assist in checking the accuracy of the input data used by the computer. The program could be made available on punched paper tape in an updated version.

C.1. References

1. Kemper, A., and Van Kempen, "A simple program for high resolution gamma ray spectroscopy"; J Rad Chem, 6:461-475; 1970.

```
 620 X(I)=(P1*SQR(B)+0.5)/T
 630 C(I)=B/T
 640 T2=T2+T
 650 IF I=1 GO TO 780
 660 IF K(I)=K(I-1)+1 GO TO 780
 670 REM CHECK FOR MISSING STEP.  TO CONTINUE AS IS ENTER 0  TO INTERPOLA
 TE 1 STEP ENTER 1.  IF THE MISSING STEP IS ON THE PEAK IT MAYBE NECESSAR
 Y TO RERUN THE SAMPLE ON THE XRAY.
 680 PRINT 'STEP MISSING BETWEEN';K(I-1);'AND';K(I);'.  ACTION';
 690 INPUT K7
 700 IF K7=0 GO TO 780
 710 K(I+1)=K(I)
 720 K(I)=K(I)-1
 730  X(I+1)=X(I)
 740 X(I)=(X(I-1)+X(I))/2
 750 C(I+1)=C(I)
 760 C(I)=(C(I-1)+C(I))/2
 770 I=I+1
 780 NEXT I
 790 GOTO 810
 800 K6 =1
 810 N7=I-1
 820 IF N7<(2*K1+7) GOTO 230
 830 T1=T2/N7
 840 REM          BACKGROUND SLOPE
 850 K3=-(C(2)+C(3)+C(4)+C(5)-C(N7-1)-C(N7-2)-C(N7-3)-C(N7-4))/(N7-6)/4
 860 K4=0
 870 REM           FIND SIGNIFICANT PEAK N4 BY COMPARING WITH STEPS A
      SEARCH WIDTH EITHER SIDE
 880 K2=K3
 890 FOR N=(N4+K1) TO (N7-3-K1)
 900 FOR I=K1+1 TO K1-1 STEP-1
 910 IF C(N)>C(N-I)+X(N) GOTO 940
 920 NEXT I
 930 GOTO 970
 940 FOR I=K1+1 TO K1-1 STEP-1
 950 IF C(N)>C(N+I)+X(N) GOTO 990
 960 NEXT I
 970 NEXT N
 980 GOTO 2170
 990 I1=N-K1+1
 1000 F=0
 1010 FOR N=I1 TO I1+2*K1-1
 1020 IF C(N)<F GOTO 1050
 1030 F=C(N)
 1040 N4=N
 1050 NEXT N
 1060 REM           FIND PEAK SHOULDERS N3 AND N5
 1070 FOR N=N4+3 TO N7-K1
 1080 IF C(N+K1)>=(C(N)-X(N)+K1*K2) GOTO 1100
 1090 NEXT N
 1100 N5=N
 1110 FOR N=N4-3 TO K1+1 STEP -1
 1120 IF C(N-K1)>=(C(N)-X(N)-K1*K2) GOTO 1140
 1130 NEXT N
 1140 N3=N
 1150 REM           DEFINE RIGHT BACKGROUND  N5 TO N6
 110 REM    PROGRAM "GKXRA" : ANALYSIS OF XRAY DIFFRACTION DATA
120 DIM K(100),C(100),X(100)
130 K6 =0
140 D2=0
150 PRINT'ENTER PROBABILITY,SEARCH WIDTH;PEAK HEIGHT ,PEAK AREA AND MASS
 OF STANDARD';
160 INPUT P1,K1,H5,S5,M5
170 OPEN 1,'XRAY2',INPUT,LINE
180 ON EOF 1 GOTO 800
190 ON ERR GOTO2200
200 L1=100
210 IF K6=1 GOTO 2220
```

```
220 N4=4
230 T2=0
240 K4=K5
250 REM          READ,CORRECT AND TRANSLATE INPUT DATA AND SEPARATE INTO
 SAMPLES
260 FOR I=1 TO 100
270 IF D2 >0 GOTO 310
280 GET L
290 IF L=L1 GOTO310
300 PRINT 'ERROR IN LINE NO ';L; ' CHECK XRAY2 ?'
310 D2 =0
320 ON ERR 1 GOTO 350
330 GET A2
340 GOTO 400
350 GET A$
360 REM CHECK FOR ERROR IN SAMPLE STEP NO. TIME COMPLEX 'A'.  IF ERROR P
RINTOUT INCLUDES ONLY A ENTER 0,A,0  IF THE COUNT IS INCLUDED IN THE ER
ROR PRINTOUT ENTER 1,A,COUNT
370 PRINT 'A=';A$;' PREVIOUS A=';A;' REVISION';
380 INPUT D1,A2,B1
390 IF D1>0 GOTO 470
400 ON ERR 1 GOTO 430
410 GET B1
420 GOTO470
430 GET  B$
440 REM CHECK FOR ERROR IN THE COUNT.  IF ONLY THE COUNT IS IN ERROR ENT
ER 0,COUNT  IF THE ERROR PRINTOUT INCLUDES THE FOLLOWING LINE NUMBER ENT
ER 1,COUNT.
450 PRINT 'COUNT=';B$;' A=';A;'PREVIOUS COUNT=';B;' REVISION';
460 INPUT D2 ,B1
470 A=A2
480 L1=L+10
490 B=B1
500 A1=A/100000
510 K5=INT(A1)
520 IF K5<>K4 GOTO 810
530 A1=(A-100000*K5)/1000
540 K(I)=INT(A1)
550 A1=A-100000*K5-1000*K(I)
560 T=INT(A1)
570 IF T>0 GOTO 620
580 T1=T2/I
590 REM CHECK FOR ERROR IN THE COUNTING TIME
   ' PRINT 'TIME IN ERROR';L;A;B;'PREVIOUS';T1;C(I-1);' ENTER TIME';
     NPUT T
 1730 IF S1<.5 GOTO 2160
 1740 S2=SQR(T+((N5-N3+1)**2)*(B1/K3+B2/K2))/A/S1/SQR(T1)*100+0.005
 1750 H=C(N4)-(B1*(N4-N2)+B2*(N6-N4))/(N6-N2)
 1760 M1=(H/H5)*M5
 1770 M2=(S1/S5)*M5
 1780 PRINT USING 1790 K4,K(N4),C(N4).K(N3),K(N5),S2,B2,K2,B1,K3,X2,Y4,H,
 M1,S1,M2
 1790: ###  *### ######.# ### ### ####.##*#####.# ## #####.# ## ####.## #
 #          NET HEIGHT######.#  MASS####.###  AREA#######.#  MASS####.###
 1800 REM OUTPUT GIVES SAMPLE NO:PEAK STEP,HEIGHT AND REGION:AREA SD: BAC
 KGROUND VALUE &NO STEPS,LEFT & RT:SLOPE & NO. OF LOOPS
 1810 REM            CHECK BACKGROUND SLOPE
 1820 Y7=(B1-B2)/(N5+N6-N2-N3)*2
 1830 Y6=ABS(Y7-Y2)
 1840 IF Y6 <(Y(N2)+Y(N6))/20 GOTO1940
 1850 REM CHECK TO SEE IF THE BACKGROUND SLOPE HAS CHANGED SIGNIFICANTLY
 DURING THE CALCULATION.
 1860 REM TO CONTINUE ON TO THE NEXT PEAK ENTER 0,0
 1870 REM TO RECALCULATE THE.PEAK HEIGHT AND AREA ENTER 1,TRUE WHERE THE
 VALUE OF TRUE IS ROUNDED OFF TOWARD SLOPE
 1880 PRINT 'SLOPE= ';Y2;'TRUE= ';Y7;' 0,0 OR 1,TRUE';
 1890 INPUT Y5,Y7
 1900 IF Y5=0 GOTO1930
```

```
1910 X2=X7
1920 GOTO1160
1930 REM  TEST PEAK WIDTH AND LIMIT IT IF NECESSARY
1940 J1=INT(12.5*H*(N6-N2)/(B1*(N4-N2)+B2*(N6-N4)))
1950 J2=0
1960 REM LIMIT THE EXTENT OF THE RIGHT EDGE OF THE PEAK.
1970 IF N5<=N4+4+J1 GOTO 2040
1980 J2=1
1990 N5=N4+4+J1
2000 J9=2*N5-N3+3
2010 IF N6<J9 GOTO2040
2020 N6=J9
2030 REM LIMIT THE EXTENT OF THE LEFT EDGE OF THE PEAK.
2040 IF N3>=N4-4-J1 GOTO2100
2050 J2=1
2060 N3=N4-4-J1
2070 J8=2*N3-N5-3
2080 IF N2>J8 GOTO 2100
2090 N2=J8
2100 IF J2=0 GOTO 2140
2110 H1=H*(N6-N2)/(B1*(N4-N2)+B2*(N6-N4))
2120 REM NOTIFY USER THAT THE PEAK WIDTH HAS BEEN LIMITED.
2130 PRINT 'WIDTH LIMITED; H1=';H1
2140 IF J2=0 GOTO 2160
2150 GOTO 1550
2160 GOTO 860
2170 PRINT USING 2180 X4,L1,C(1),X(1),C(N7),X(N7),T1
2180: ###    NO MORE PEAKS   LAST LINE IS###### #####.# ## ####.# ##T@##
#.#  ***
2190 GOTO 210
2200 PRINT 'ERROR TYPE  ';ERM;'  IN SAMPLE ENDING ON LINE  ';L1
2210 GOTO 210
2220 CLOSE 1
2230 END


1160 X4=X4+1
1170 F=0
1180 FOR N=N5 TO 2*N5-N3+3
1190 F=F+C(N)-X2*(N-N5)
1200 Z=F/(N-N5+1)
1210 IF N+1>N7 GOTO 1260
1220 IF Z>=(C(N+1)-X(N+1)-X2*(N-N5+1)) GOTO 1250
1230 IF N+2>N7 GOTO 1250
1240 IF Z<(C(N+2)-X(N+2)-X2*(N-N5+2))GOTO1260
1250 NEXT N
1260 N6=N
1270 F=0
1280 FOR N=N6 TO N4+1 STEP -1
1290 F=F+C(N)+(N6-N)*X2
1300 Z=F/(N6-N+1)
1310 IF Z>=C(N-1)-X(N-1)+X2*(N6-N+1) GOTO 1330
1320 IF Z<C(N-2)-X(N-2)+X2*(N6-N+2) GOTO 1340
1330 NEXT N
1340 N5=N
1350 REM          DEFINE LEFT BACKGROUND N2 TO N3
1360 F=0
1370 FOR N=N3 TO2*N3-N5-3 STEP -1
1380 F=F+C(N)+X2*(N3-N)
1390 Z=F/(N3-N+1)
1400 IF N<2 GOTO 1450
1410 IF Z>=(C(N-1)-X(N-1)+X2*(N3-N+1)) GOTO1440
1420 IF N<3 GOTO 1440
1430 IF Z<(C(N-2)-X(N-2)+X2*(N3-N+2)) GOTO 1450
1440 NEXT N
1450 N2=N
1460 F=0
1470 FOR N=N2 TO N4-1
```

```
1480 F=F+C(N)-Y2*(N-N2)
1490 Z=F/(N-N2+1)
1500 IF Z>=(C(N+1)-Y(N+1)-Y2*(N-N2+1)) GOTO 1520
1510 IF Z<(C(N+2)-Y(N+2)-Y2*(N-N2+2)) GOTO 1530
1520 NEXT N
1530 N3=N
1540 REM          CALCULATE BACKGROUND
1550 Y3=N6-N5+1
1560 F=0
1570 FOR N=N5 TO N6
1580 F=F+C(N)
1590 NEXT N
1600 B1=F/Y3
1610 Y2=N3-N2+1
1620 F=0
1630 FOR N=N2 TO N3
1640 F=F+C(N)
1650 NEXT N
1660 B2=F/Y2
1670 REM          CALCULATE AND PRINT RESULTS
1680 T=0
1690 FOR N=N3 TO N5
1700 T=T+C(N)
1710 NEXT N
1720 S1=T-(B1*(N5-N2)+B2*(N6-N3))*(N5-N3+1)/(N6+N5-N3-N2)
```

APPENDIX D

PREPARATION OF ROCK SAMPLES FOR ANALYSIS

During these studies, samples of rock dust were prepared for comparing with the samples of airborne dust to assist in understanding the compositional changes and interference effects. A simple liquid dispersion technique was developed to give even deposition over the filter and was found to give reasonable agreement between X-ray analysis for quartz and the free silica analyses obtained from mine sources. It is realized that this simple technique could suffer errors from two sources — preferred orientation and mineral segregation by size in the settling process, but to date these have not appeared to give rise to significant error in quartz analysis. However, other minerals can give markedly different intensity ratios for the various diffraction lines when prepared by this method compared with the preparation method for standards; mixing with resin and setting in a recess in the sample holder. No efforts have yet been made to solve these problems; their extent is shown in Table D.1 where the calibrations for standards of various pure minerals are given.

The liquid dispersion technique gives an even dust deposit and lower X-ray intensity/mass ratios than samples collected by the personal dust sampler, and thus a different calibration is required as was given in Table B.2.

Preparing rock samples for X-ray diffraction analysis requires the following steps:

1. Handgrind 0.1 ± 0.01 gm of dust in a pestle and mortar under ethyl alcohol for 10 to 15 minutes.
2. Wash the groundrock into a 50-ml bottle with alcohol and make up to about 40 ml.
3. Place a tared 25-mm diameter 0.8-µm pore-size silver membrane filter in a specially made filter funnel, 22 mm internal diameter and 50 mm high on a smooth backing plate and seal with an O-ring.
4. Place distilled water to a depth of 25 mm in the filter funnel and nearly fill with alcohol. The use of a denser solvent minimizes density convection and uneven deposition on the filter.
5. Shake up the ground material in the bottle and let settle for one minute.
6. Remove 2 ml of suspension from near the top of the bottle using a pipette and transfer it to the funnel.
7. Turn on vacuum under funnel.
8. Remove vacuum when funnel has drained, i.e., do not wash down.
9. Dry and weigh the filter.

It is essential that even dispersion be obtained over the surface of the 22-mm dust deposit on the filter and this is difficult to achieve with dense minerals. An alternative technique using 13-mm diameter filters has been tried, but samples that are heavy enough to weigh accurately require substantial corrections for absorption. Although these can be obtained from measurements of the intensity of the silver 1,1,1 line through the dust deposit and from the back of the filter, allowance has to be made for the uncovered portion of the filter surrounding the dust deposit as well as for variations in X-ray intensity over the dust deposit. Calibration is complex and time-consuming.

Table D.1 - Calibration of pure mineral standards: usable diffraction lines

	d-spacing Å	Calibrated mass on standard, mg			d-spacing Å	Calibrated mass on standard, mg
Sulphides				Carbonates		
Chalcopyrite				Dolomite		
	3.03	3.8			2.88	2.7
	1.85	5.4			1.80	0.6
	1.59	6.5			1.78	1.1
	1.08	6.9			1.39	3.0
Pentlandite		St.I	St.III*	Siderite		
	5.84	0.42			3.60	4.7
	3.04	0.47	0.68		2.80	2.8
	1.94	0.99	0.58		2.14	5.1
	1.78	0.72	0.71		1.97	3.2
	1.31	0.53	0.76		1.74	4.0
Pyrite					1.36	5.5
	3.12	2.3				
	2.70	1.8		Silicates		
	2.42	2.6		Albite	6.39	1.45
	2.21	2.9			4.03	3.9
	1.92	2.2			3.78	3.6
	1.63	2.8			3.67	5.0
	1.04	2.3			3.19	1.9
Pyrrhotite				Chlorite		
	3.00	2.5			14.0	4.4
	2.65	4.4			7.0	2.75
	2.08	4.0			4.7	2.0
	1.73	4.2			3.5	1.8
	1.62	3.9			2.85	2.8
Sphalerite	3.12	3.8			2.0	3.5
	1.91	3.9		Labradorite		
	1.63	4.5			6.4	4.2
					4.0	3.2
Oxides					3.6	2.3
Magnetite	4.84	2.2		Microcline		
	2.96	2.2			4.2	4.0
	2.53	2.6			3.77	1.6
	2.09	2.1		Muscovite		
	1.71	3.2			10.0	1.7
	1.61	3.2			5.0	1.8
	1.12	3.3			4.5	4.3
	1.09	3.3			3.32	1.5
Uraninite					3.2	1.9
	3.15	3.5			3.0	3.0
	2.73	5.4			2.56	3.2
	1.93	5.3			2.13	2.9
	1.65	5.3		Pyroxene		
	1.58	3.8			3.04	4.8
	1.25	5.2			2.5	3.5
	1.11	4.8			1.62	5.8
	1.05	6.1				
	0.93	7.3				

*More than one attempt at preparing standards was required for some materials. It so happened there were two good standards for pentlandite.

APPENDIX E

VARIETIES OF FREE SILICA

Free silica, or silicon dioxide, SiO_2, occurs in a variety of crystalline and non-crystalline polymorphs. All these forms appear to be substantially more hazardous to health than most other minerals and must be considered in assessing respirable dust. The polymorphs of silica with their occurrences and properties have been described in detail (1). Free silica occurs chiefly in the form of quartz. Crystobalite and tridymite occur occasionally in younger rocks. Semi-amorphous forms such as opal and amorphous forms such as glasses can be found in nature. Quartz occurs in many varieties depending on origin and impurities.

Because of the complexity and variety of free silica in nature it is desirable to check the X-ray response to many forms rather than to compare all samples with a particular variety.

The Geological Survey of Canada and other sources supplied a number of mineral samples of nearly pure quartz in a variety of forms as well as samples of crystobalite and pure synthetic tridymite. One sample of crystobalite was scraped off an obsidian matrix. Samples for X-ray analysis were prepared using the technique described in Appendix D. The minerals are described in Table E.1 and the relative X-ray intensities compared with the standard materials used for each crystalline form are given in Table E.2.

Dana classifies quartz into two main classes — "coarse crystallized" and "fine crystalline to dense" (1). Samples 1 and 2 are examples of the first class and are in very close agreement. Samples 3 to 11 are fine crystalline to dense varieties. Sample 3, novaculite, shows good agreement with the coarse crystalline varieties except for the strongest quartz line, 1,0,1, which is about 20% lower in intensity. The reduction on this one line is also found on the Jasper sample. The other fine crystalline varieties, 4 to 11, show generally weaker intensity/mass ratios and variations in intensity between the diffraction lines. Jasper is the

Table E.1 - Description of free silica samples

Code No.	Description	Origin
1	Laboratory 'standard' quartz	
	Silica sand	Ottawa, Illinois
	Quartz varieties	
2	Massive white	Elliot Lake, Ontario
3	Novaculite	Hot Springs, Arkansas
4	Jasper	Pink Lake, P.Q.
5	Chalcedony	Kamloops Lake, B.C.
6	Chalcedony	Tranquille Lake, P.Q.
7	Chalcedony	Red Deer, Alta.
8	Agate centre	Western U.S.A.
9	Agate edge	Western U.S.A.
10	Flint	Dover Cliffs, U.K.
11	Chert	Mogul mine, Eire
12	Prepared respirable quartz DQ12	Dr. Robock, Germany*
13	Prepared respirable quartz GQ12	Dr. Robock, Germany*
14	Crystobalite	Valleyview, Alta.
15	Crystobalite in obsidian	Millar County, Utah

*See reference 2.

Table E.2 - X-ray intensities of silica varieties

Code		X-ray diffraction line			
		1,0,0	1,0,1	1,1,2	2,1,1
	Quartz				
	Intensity[1]/mass ratio for each variety compared with Type 1				
2	Massive white[2]	1	1	1	1
3	Novaculite	1.04	0.82	0.97	1.03
4	Jasper	0.74	0.61	0.71	0.80
5	Chalcedony	0.62	0.60	0.86	0.76
6	Chalcedony	0.39	0.57	0.68	0.54
7	Chalcedony	0.55	0.67	0.79	0.75
8	Agate centre	0.52	0.65	0.81	0.65
9	Agate edge	0.53	0.64	0.79	0.66
10	Flint	0.62	0.65	0.81	0.79
11	Chert	0.63	0.59	0.81	0.81
12	Prepared	--	0.72	0.50	0.57
13	Prepared	--	0.74	0.62	0.62
	Crystobalite				
	Intensity/mass ratio for 15 compared with 14				
15		0.79	0.36	0.40	0.44

[1] X-ray intensity determined as area of the diffraction peak.

[2] Within ±5% on three samples.

only material in this group reputed to have more than 10% impurities — non-quartz material — which was confirmed by the X-ray scan. If allowance is made for the amount expected, results are close to those obtained for Novaculite. The chalcedonies, agates, flint and chert, although reputedly composed of more than 90% quartz, may contain inclusions of impurities (1). However, only in the case of chert is sufficient crystalline impurity indicated by X-ray diffraction scans to account for the lower intensity/mass ratio.

It appears that the 1,1,2 line gives the highest and most consistent estimate of the fine-grained varieties, but this is difficult to apply because of the effects of interference.

Samples 12 and 13 are of quartz dust prepared by grinding and elutriation to have a particle size of less than 5 µm and are intended for biological experiments on silicosis. These show low X-ray intensity/mass ratios which may be attributed to small particle size, surface decrystallization or impurities. Examination of industrial products prepared in Canada from massive white quartz suggested that the sericite intensities found in it were due to an impurity of less than 1% in the ore being concentrated into the fine fraction. It formed about 30% of the dust less than 2 µm in size as determined by the non-quartz discrete lines in the X-ray diffraction pattern. While the concentration of silicate impurities into the fine DQ12 fraction of the German quartz sand would be expected, this is not confirmed by the German analyses or by CANMET X-ray diffraction scans.

Two samples of crystobalite were examined and found to have large differences in the X-ray intensity/mass ratios. This is believed due to the inclusion of obsidian in sample 15 because of difficulties in separation.

A sample of tridymite was found to give a complex pattern with low intensity/mass ratios;

a much simpler pattern with much higher ratios should be found if it is heated above the phase change at 163°C to form high tridymite and thus a heated specimen holder is required for analyzing airborne dust containing tridymite (3).

This very limited study has demonstrated that large differences in X-ray intensity/mass ratio of up to 2:1, can arise with different samples of quartz. Although this is partly due to impurities, there seems to be a remaining real effect possibly as great as 40%. This needs to be borne in mind in considering the health hazard due to airborne dust. Most, but not all, occurrences of significant quantities of quartz in airborne mine dust arise from coarse crystalline sources and it is fortunate that the X-ray diffraction method gives a reasonable consistent assessment. Fine crystalline varieties of quartz can occur in significant proportions in some mines and the possibility of underestimating the quartz content using X-ray diffraction should be considered.

E.1. Conclusions

1. There are differences between X-ray intensity/mass ratios for different varieties of quartz.
2. Coarse crystalline quartz is most common and in most cases should be used for calibration of the X-ray equipment.
3. Special studies should be undertaken for each mine with significant quantities of fine crystalline quartz, i.e., chert or other silica varieties. These studies should measure the X-ray intensity/mass ratios and the purity of hand picked samples of the purest possible free silica.
4. Because of uncertainties about impurities in fine crystalline quartz and their biological effects it is suggested that fine crystalline quartzes be expressed in terms of equivalent coarse crystalline quartz as given by the analytical method.

E.2. References

1. Frondel, C. "Dana's system of mineralogy"; III; Wiley, New York; 1962.

2. Robock, K. "Standard quartz DQ12<5 μm for experimental pneumoconiosis projects in the Federal Republic of Germany"; Ann Occ Hyg; 16:63-66; 1973.

3. Borg, I.Y. and Smith, D.K. "Calculated X-ray powder patterns for silicate minerals"; Geol Soc of Am; Boulder, Colorado; 1969.

APPENDIX F

CALIBRATION OF CHARACTERISTICS OF SIZE SELECTIVE DUST SAMPLERS

During these studies it was found that commercial dust samplers intended to measure respirable dust to the same specification, could give significantly different estimates of concentration under certain test conditions as summarized in Table F.1 (1). These were traced to a number of causes such as variations in dimensions and filter holders and it was concluded that calibration of the size selection characteristics of the completely assembled sampler rather than its size selection component alone is essential.

The basic requirements of any measuring instrument are accuracy, repeatability and reliability. For two-stage dust sampling instruments intended to simulate the collection of dust in the two main regions of the lungs, one of the major factors is performance of the size selector in separating the dust into two parts. Three specifications for the size selector have been set down by the British Medical Research Council (2,3), by the U.S. Atomic Energy Commission (4), and by the American Conference of Governmental Industrial Hygienists (5) — a modification of the second specification.

It has been shown that the theoretical difference between these three specifications would be about 20% in most polydisperse industrial dust clouds (6,7). This is less than the difference between individual persons or for one person using different breathing patterns.

The first specification has the advantage for reproducibility of specifying a size selection curve based on a calculable physical process horizontal elutriation, and approximately to a lung curve (8). The other two are based on a mean lung curve to which the size selection curve of real instruments can only be approximately matched by a difficult and time-consuming laboratory calibration. Size selective sampling has been reviewed (9,10). An instrument for use with the first specification has been adopted as a standard in both the U.K. (11) and the U.S.A. (12, 13).

The horizontal elutriator size selector, however, has to be used in a position close to horizontal and is not suitable for personal sampling. Cyclone size selectors can be used in any position and although they do not give the same curve as a horizontal elutriator, instruments fitted with them can be adjusted to indicate approximately the same respirable dust concentration (14). This approach should give more reproducible measurements than using a mean factor to correct from one size selection curve to another in dust clouds of differing size distribution as has been done in the U.S.A. (12,15). The latter procedure has been criticized as giving a very poor prediction of the concentration measured by the instrument on which the dust standard is based (15).

A second important criterion is the flow rate through the dust sampling instrument. With the horizontal elutriator, correct flow rate is very important as a 10% increase in flow leads to a 20% increase in mass of dust collected on the filter. With a cyclone, flow control is not as important because a 10% increase in flow has hardly any effect on the mass collected. This is because of the compensating effects of flow rate and collection efficiency of the cyclone (14). Of course, in very fine dust clouds the mass collected is proportional to flow rate for both types of size selector.

Most small air samplers use battery-powered single action pumps producing a pulsating airflow that affects the size selection characteristics (15) and indication of airflow by a taper tube flowmeter which tends to read the root mean square rather than the average flow. It is thus desirable that the airflow in all instruments should be sufficiently smooth to give a negligible error in size selection and flowmeter reading which are supplied by manufacturers with a calibration based on smooth airflow. The pulsating airflows from single action pumps can be

Table F.1 - Performance of size selector - dust sampler

Size selector	Dust sampler	Flow		Relative[2] respirable dust concentrations
		L/min	Code[1]	
10-mm nylon cyclone	Lab A-E	1.7	C	0.74 - 0.84
	F	1.7	C	0.82
	F	1.7	PS	0.74
	Man 1	1.7	P	0.63
	Man 2 A	1.7	P	0.49
	" "	1.7	PS	0.67
	" B	1.7	P	0.41
	" "	1.7	PS	0.56
	" "	1.7	C	0.55
	" "[3]	1.7	PS	0.79
	" "	1.7	C	0.81
	" "[4]	1.7	PS	0.77
	" "	1.7	C	0.79
3/8-in. metal cyclone	Man 3 A	2.0	PS	0.76
	" B	2.0	PS	0.85
	" C-J	2.0	PS	0.92 - 1.06
	" K[5]	2.0	PS	1.21
	" L[5]	2.0	PS	1.13
	" M-P	2.0	PS	1.0 - 1.08
Horizontal elutriator	Lab A	2.83	C	1.0
	" [6]	2.83	C	0.94
	" [7]	2.83	C	0.86
	Man 3 Q	50.00[8]	C	0.95
	" R-T	2.5	P	0.98 - 1.05
	" R	2.5	PS	0.99 - 1.02
	" "[9]	2.5	C	0.97 - 1.01

[1] Code for evenness of flow: C - critical orifice (very smooth), P - pump, PS - pump and smoother.

[2] Dust concentration relative to that given by the Hexhlett in a coarse coal dust cloud. External flow control used and internal critical orifice removed.

[3] Commercial unit with alternative filter holder.

[4] Commercial unit with laboratory fitted metal filter holder.

[5] Some dimensions on these 2 cyclones were outside reasonable tolerances.

[6] Laboratory unit with filter facing down (Reference 1).

[7] Laboratory unit with small orifice between elutriator and filter.

[8] Hexhlett as manufactured with critical orifice between elutriator and filter.

[9] Cassella 113A as recommended for standard instrument.

smoothed using pulsation dampers (Appendix G).

A third factor in these instruments is electrical conductivity of the components as this may affect deposition of charged particles. The mucous fluid in the bronchial passages is moderately conducting in common with other physiological fluids. Dust deposition on plastic components of dust samplers, attributed to electrostatic effects has been found in tests in this laboratory under dry conditions. This deposition has been associated with a 20% lower estimate of respirable dust in a plastic filter holder compared with a metal holder.

A fourth factor is attaching the size selector to the filter. It was found that dust can deposit on intermediate surfaces causing changes in the effective size selection characteristics.

A fifth factor is the type of filter used. It is widely accepted that a relatively coarse filter of 5-μm pore size or equivalent will collect airborne dust from comminution with high efficiency but that a finer pore size may be required for smokes or fumes.

A sixth factor is the direction and magnitude of the air stream passing over the dust sampler. It has been shown that the dust collected by the 113A dust sampler is dependent on airflow direction and velocity (16). This is also probably true of other dust sampling instruments and of the human nose and mouth (17).

Comparison tests between instruments should be carried out at low air velocities of about 0.5 m/s to avoid inertial effects. Although velocity and direction effects on each sampler should be determined, corrections cannot readily be applied because little is known about their effects on nasal entry and dust deposition in lungs.

F.1. Dust Collection Specifications

For proving instrument types, accurately calibrated airflow equipment such as a spirometer or wet test gas meter is required. However, for checking individual instruments of an approved design, calibrated high-accuracy glass rotameters provide satisfactory secondary standards.

1. Standard size-selection specification. The gravimetric sampler designed by the Mining Research Establishment of the National Coal Board, U.K., and manufactured by Casella uses a horizontal elutriator to the BMRC specification and is readily available. This design was chosen as a standard in the Elliot Lake Laboratory. For critical work, such as setting up and proving other instruments, a much closer specification on airflow is required; a flow rate of 2.5 ± 0.025 L/min from a critical orifice with the filter holder and horizontal elutriator is recommended. Checks should also be made to ensure that the leakage is very low.
2. All other respirable dust samplers should be so adjusted as to choice of airflow, etc. that the estimated respirable dust concentration based on the average of at least 5 instruments should be within 5% of that of the standard instrument in a 'test' dust cloud. The test dust cloud is dispersed from dry pulverized coal by an air ejector. The dust should be 40-60% respirable by mass.
3. The airflow through the size selection device and filter holder should be smooth enough for the dust collection to be within 5% of that obtained with a smooth airflow.
4. Components of samplers from each manufacturer should have equal ability within 5% of collecting dust in the test cloud, and be numbered in series each instrument kept intact or retested when components are changed.
5. The cyclone and filter should be constructed of materials having surface conductivities greater than 10^{-6} S/cm to avoid build-up of electrostatic charge and possible interference with dust deposition.
6. Individual instruments are considered satisfactory if they agree within 10% of the

standard in the test dust cloud.

7. Airflow from a pump and damper must be smooth enough to give less than 5% error on a rotameter calibrated on a smooth airflow. Note, however, that for cyclone size selectors, specification 3 is approximately equivalent to a 2% error in this test.
8. The flow rate should not drop more than 10% after collecting 10 mg/m³ of the test dust in an 8-hr shift when the flow is set 5% higher at the start of sampling and no further adjustment of the flow controls is made.
9. Test procedures and standards should be established for each instrument to determine
   i) leakage, including inlet fitting,
   ii) pressure drop and flow rate,
   iii) pump and valve maintenance.
10. Calculation of respirable dust concentrations should be based on:
    i) nominal flow rate for cyclone size selectors rather than by using the mean of before and after flows,
    ii) mean of flow before and after, for horizontal elutriator size selector,
    iii) mass airflow relationship found in test dust cloud for other size selectors.
11. The pumping performance of a dust sampler can depend on air density. The effect of operating it at pressures experienced in a deep mine should be determined and an appropriate adjustment made to the flow rate established on surface prior to sampling and calculated to minimize error at depth in the mine.

F.2. Test Methods

Test dust cloud

Dry coal is crushed and then ground in a rotating plate pulverizer with plates set at 0.125-mm separation. It is dispersed using an air ejector at a pressure of 60 kN/m² (9 lb/in.²). The dust cloud should have a concentration of 10-30 mg/m² and contain between 40 and 60% respirable dust by mass.

Dust collection

Standard size selection

The 113A gravimetric dust sampler as manufactured by Casella is used as the standard instrument (18). When testing it is necessary to adjust the flow rate as accurately as possible; however, this is easier to achieve if the elutriator and filter holder are operated on a smooth airflow produced by a vacuum pump acting through a critical orifice rather than by the instrument pump.

The Hexhlett dust sampler is useful as a secondary standard because of its large sampling rate of 50 L/min and correspondingly low weighing errors (18) GF/A filters of 5.5 cm in diameter are used. The Hexhlett indicates a respirable dust concentration between 0.85 and 1.0 times that of the standard instrument depending on the flow control used. The use of the critical orifice supplied leads to a low estimate of respirable dust because of dust deposition on metal surfaces between it and the filter. The mass of dust collected by the standard and test instruments should be about 2 mg.

Tests with pump or smooth flow
(9.1. para 3)

A sampling head with filter holder is alternately tested in the test cloud with airflow produced by its own pump, including a pulsation damper, and with a smooth flow derived from a critical orifice (19). The smooth airflow has to be carefully adjusted using a wet test gas meter or similar meter to be equal to the pump flow.

Airflow

Accuracy of rotameter reading on pump flow

The wet test gas meter, or similar instrument, and the rotameter are set up in series and air is drawn through them with either the instrument pump including pulsation damper or a critical orifice and vacuum pump (Fig. F.1). The difference in rotameter reading on the pump flow with its pulsations and that given by a critical

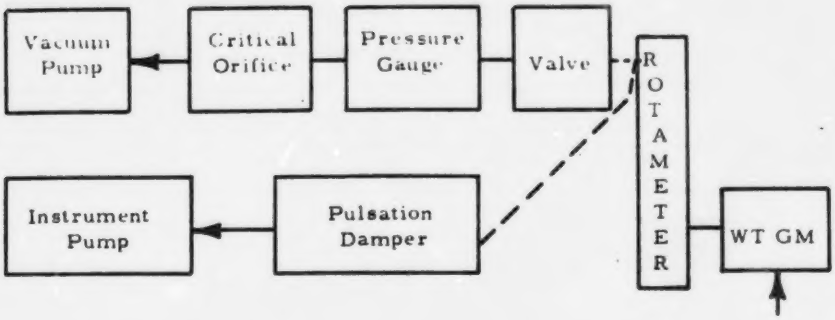

Fig. F.1 - Accuracy of rotameter reading on pump flow test.

orifice adjusted to the same flow rate as indicated by the wet test gas meter gives a measure of whether the pulsations are sufficiently smoothed by the pulsation damper or not.

Flow rate control

The instrument flow rate is set at 5% above nominal and then collects dust in the dust chamber for eight hours at a rate of 8-12 mg/m³. The final flow rate is measured and the flow rate drop corresponding to a dust concentration of 10 mg/m³ is calculated on the assumption that the measured drop is linearly proportional to the respirable dust concentration measured by the instrument. Alternatively this can be determined from separate measurements of pressure drop on the filter and the effect of pressure drop on the airflow of the pump.

(i) Leakage tests

Leakage tests should be carried out on the complete unit excluding the pump, making it essential to have a leakproof fitting attached to the sampler inlet. The test layout is shown in Fig. F.2. Leakage is indicated by change in rotameter reading as valve 1 is adjusted from fully open to a gauge pressure drop of 2.5 kPa.

(ii) Flow and pressure drop

Figure F.3 shows layout for testing the pump over its complete pressure range. A useful test point is the suction developed at a flow 10% low after the pump is set to 5% high on a clean filter because this is considered the limit on a valid sample; another useful point is the maximum suction developed as the inlet is closed. The motor current should also be measured.

(iii) Maintenance procedures

Specifications on the performance of the units in tests 9(i), (ii) should be set up to indicate the need for pump and motor maintenance. A daily schedule should be set up to indicate the need for more complete testing and maintenance as outlined above.

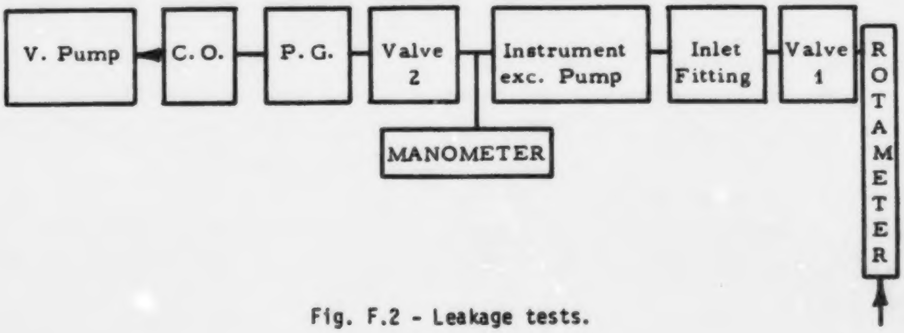

Fig. F.2 - Leakage tests.

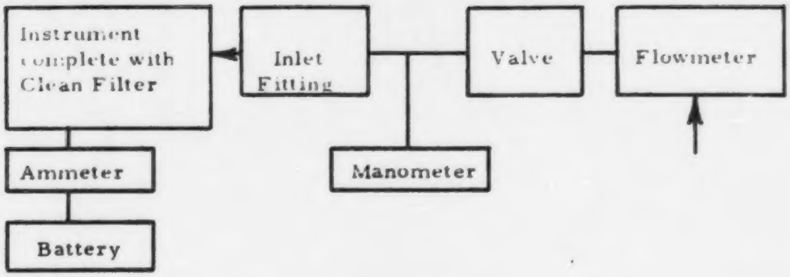

Fig. F.3 - Flow and pressure-drop test.

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APPENDIX G

DEVELOPMENT OF THE CANADIAN MINING PERSONAL DUST SAMPLER (CAMPEDS)

Most studies carried out by the Elliot Lake Laboratory in mines used the Casella 13040 personal size-selecting gravimetric dust sampler (1,2). Although this instrument has proved capable of collecting samples suitable for gravimetric and direct quartz analyses using X-ray diffraction on the filter some problems have occurred:

(a) flow rate adjustment by bleed valve was a source of dust entering the pump and increased the need for maintenance;

(b) flow smoother was bulky, awkward, and leaked frequently;

(c) the instrument is too fragile as a personal sampler in mines;

(d) miners are reluctant to carry extra equipment.

However, these problems have been overcome through a number of improvements.

It is believed unnecessary to adjust airflows underground because the mass of respirable dust collected is almost independent of airflow except in dust clouds with unusually high proportions of fine particles (3). If it were possible to adjust the airflow underground, it seems unlikely it would be carried out sufficiently systematically by mine staff in routine use to be of any value.

Tests on flow smoothers led to a much smaller and convenient design; specifications are given in Appendix F.

The problem of the fragile dust sampler could have been met by using a more robust and heavier case, but was approached by combining the sampler with a piece of existing miners' equipment. Its combination with cap lamp and battery terminal, SIMPEDS, was developed by the Safety in Mines Research Establishment (4,5). This showed promise but could not collect samples for direct quartz analysis on the filter because of the coarse pre-filter used to catch any of the dust layer dislodged by rough handling. The SIMPEDS is also cumbersome to wear because of the extra hose and cap lamp-mounted sampling head.

Making the sampler as inconspicuous as possible by combining both the sampling head and pump with the cap lamp battery was proposed and tests were made (Section 5.2).

The concept of combining the dust sampler with the cap lamp battery is termed the Canadian Mining Personal Dust Sampler with its acronym, CAMPEDS. The first prototype was made using standard Casella 13040 personal sampler components mounted in a metal case to fit the top of the Wheat battery, as shown in Fig. G.1. These prototypes have been operated by mine staff under the direction of the Mines Accident Prevention Association of Ontario (6). Although wearing the units was voluntary, few refused (7). The main problem has been changing filters between shifts, because the men available for this purpose had no previous laboratory experience (7). The sampling head was designed so that the filter could not be handled when exposed but it has occasionally been dropped and damaged. This problem led to a second prototype using a smaller impaction size selector instead of the cyclone, following the suggestion of M. Jacobsen (8,9), making it a part of the reloadable cassette as shown in Fig. G.2. This prototype uses the SIMPEDS pump unit with minor modifications.

The Casella SIMPEDS pump used in the CAMPEDS is for mounting on the Oldham cap lamp battery but both the MSA and the Wheat cap lamp batteries can be machined to fit it. In laboratory tests both types of battery were hand filed to fit.

G.1. Impaction Size Selector

The size selector combined with a filter holder for 25-mm diameter filters is shown in Fig. G.2.

The first model used a single impaction orifice, but it was found that on heavy dust loads of about 2 mg, an oversize dust cone built up under the orifice and that parts of this could

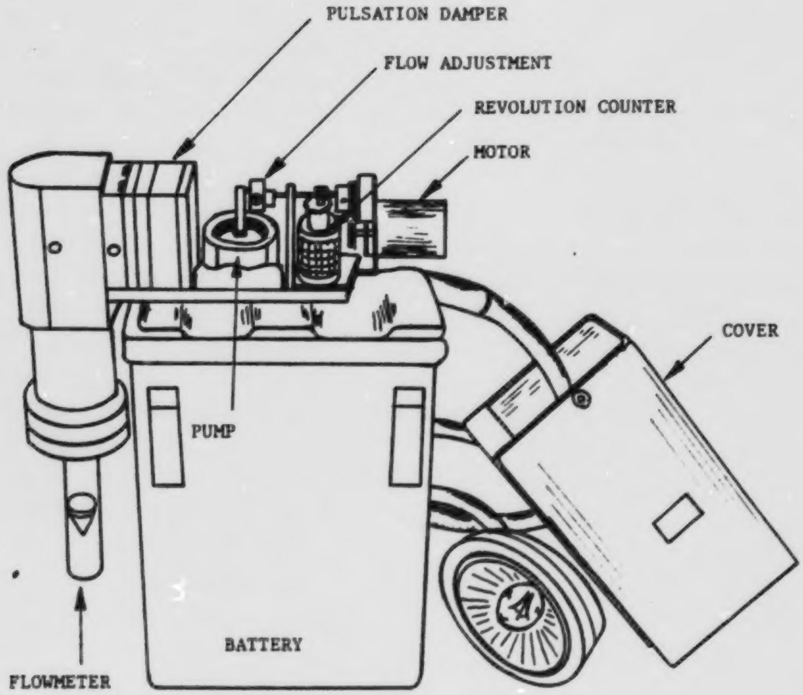
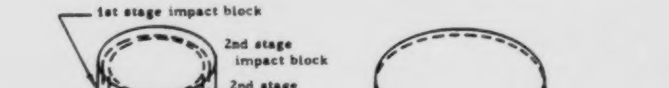

PULSATION DAMPER
FLOW ADJUSTMENT
REVOLUTION COUNTER
MOTOR
COVER
PUMP
BATTERY
FLOWMETER

Fig. G.1 - The pump set up for adjusting flow rate.


1st stage impact block
2nd stage impact block
2nd stage orifice
O-ring
Cone
Washer
Filter
Support grid
O-ring
Filter holder
Air outlet
Air entry
1st stage orifice
Cassette cover
Battery clamp
Mounting position
Modified SIMPEDS pump and case
Air entry
CAP LAMP BATTERY

Cassette - exploded view

Assembled sampler

Fig. G.2 - Filter holder and size selector for the CAMPEDS.

be readily dislodged and pass through to the filter. The problem was solved by using two orifices in series with the first only slightly larger than the second so that most of the dust was trapped there. The suggestion (9) of using a lip, although possible with this construction, was not practicable because of the difficulties of cleaning behind it. Impact surfaces are lightly coated with vaseline to decrease the possibility of coarse dust penetrating to the filter. The flow rate chosen for this sampler was 2 L/min as a round figure within the range of the specially built Rotameter taper-tube flowmeters supplied by Casella. Some consideration was given to the flow of 2.08 L/min which would give the exact volume of 1 m^3 in an 8-hr period normally used to define the period of exposure for threshold limit values, but it is slightly too high for convenience with the high accuracy flowmeter custom-manufactured for Casella.

Circular impaction orifices were chosen for the size selector for ease of construction and ready checking. The diameter of these orifices was adjusted so that the completed unit, when operated at 2 L/min, indicated the same respirable dust concentrations as the 113A dust sampler, as described in the section on calibration in Appendix F. The effect of orifice size is shown in Table G.1 both for single orifices and two in series.

Tests were carried out on size selection characteristics of the final CAMPEDS impactor and a horizontal elutriator, the 113A, designed to the BMRC specification (10). The technique used is similar to that described by Tomb and Treaftis (9). The Coulter counter, an electronic size analyzer classifying particles by volume, was used to compare coal-dust samples collected on silver membrane filters following the various selectors with those collected on an open filter in the laboratory test chamber. The dust samples were redispersed in ethyl alcohol and an aliquot transferred to the electrolyte, 4% sodium hexametaphosphate, for counting. The detailed results are given in internal reports and summarized herein. The number of particles in each size range for some of the experiments are shown plotted against size on an approximately logarithmic scale in Fig. G.3. These results were obtained using two Coulter counter aperture sizes. Figure G.4 shows results obtained from percentage penetration through the respirable-dust size selectors plotted against particle size by volume. Also shown is the design performance plotted against aerodynamic particle size. It can be seen that the results are not very consistent, presumably due to difficulties in removing all the dust from the filter, dispersing the dust, and avoiding contamination. These experiments show no significant differences between the CAMPEDS and the horizontal elutriator size selectors, but they are not accurate enough to detect the expected slight differences in slope.

G.2. Laboratory Tests on CAMPEDS Sampler

The SIMPEDS 70 Mark 2 flow smoother and flowmeter was found to have a high resistance to airflow — about 10 mm of mercury at 2.0 L/min. This left little spare capability for drawing air through the size selector, filter, and dust deposit, especially when a 25-mm diameter filter is used instead of the specified 37 mm. Resistance of the flow smoother was greatly reduced by opening up the outlet to 4.0 mm, the hole between the two chambers to 2.5 mm, and removing the 30-mm long by 1.3-mm diameter orifice in the smoother inlet. It was then found that tests 9.3.3 and 9.3.7 were satisfactory and that a gauge pressure of 2.5 kPa across the filter was equivalent to a drop in flow rate of 10%. If this is not sufficient to handle the dust load in mines with high diesel usage, it will be necessary to try the unit without the built-in flowmeter or to modify the size selector and use a lower flow rate.

Air density increases with mine depth and this affects flowmeter calibration — the reading is about 10% high at 2000 m below surface. It is possible that the pump performance is also affected. A temperature effect was noticed in the mine tests. Therefore an experiment was carried out in the laboratory in which

Table G.1 - Effect of diameter and number of orifices on the characteristics of an impact size selector

Diameter of orifice mm		Indicated respirable dust concentration relative to that of the 113A
1st	2nd	
NU*	4.4	1.68
NU	3.2	1.25
NU	2.5	1.5
4.4	4.1	1.34
4.4	3.8	1.28
4.4	3.2	0.93
3.8	3.2	0.88
3.8**	3.5**	1.00

*Not used.

**Values chosen for the CAMPEDS dust samplers.

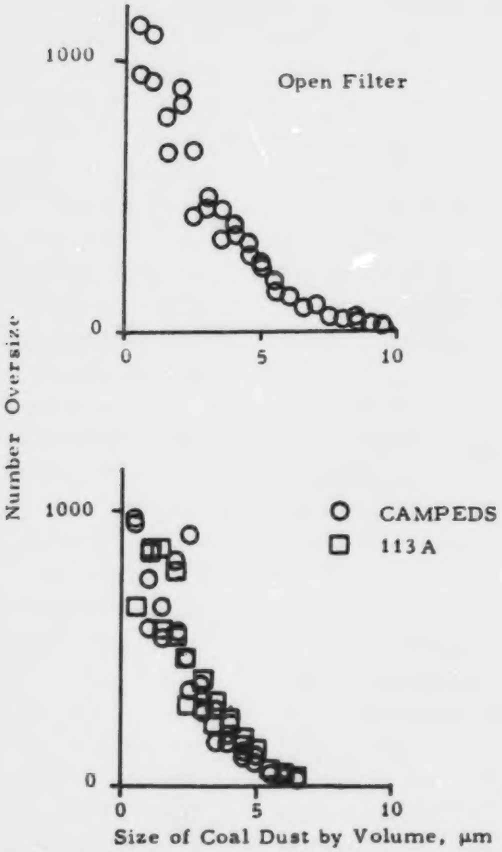


Fig. G.3 - Size distribution of particles determined by Coulter Counter.

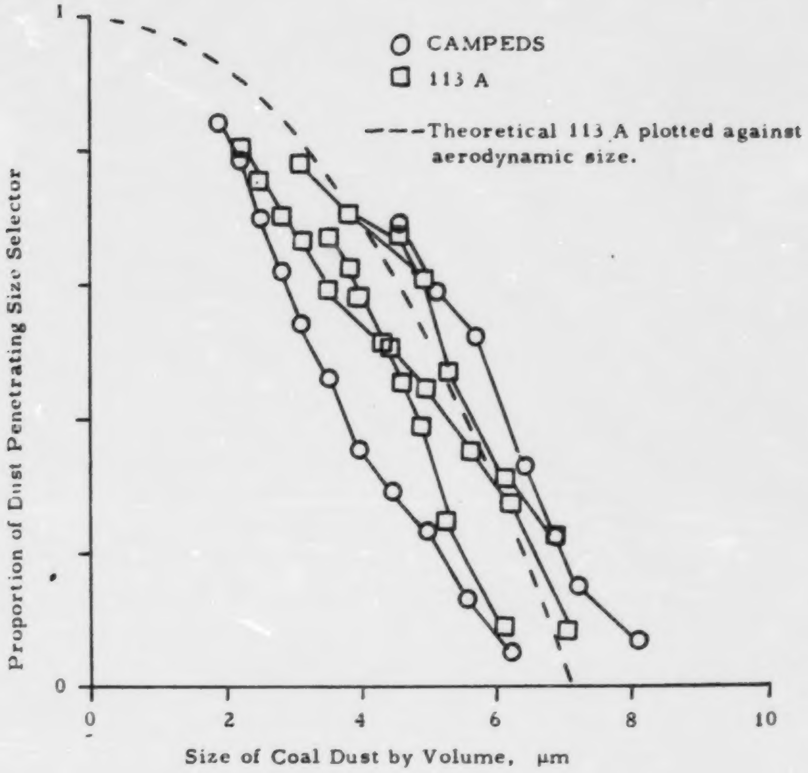


Fig. G.4 - Efficiency of collection of particles by size selectors determined by Coulter Counter.

two CAMPEDS units were assembled in a pressure chamber and measurements were made over a range of pressures under two temperatures. Figure G.5 shows the results obtained for motor speed (expressed as true seconds per indicated minute on the running time meter), for rotameter reading and for volume flow obtained by correcting the rotameter reading to test pressure and temperature.

It can be seen that increasing pressure leads to decreased motor speed, increased indicated flow, and decreased true volume under test conditions. Qualitatively this is expected because of increased air density increasing the load on the pump and the rotameter indication.

Lowering the temperature by 20°C reduced airflow slightly and altered motor speed by about 2% on each unit but in opposite directions.

It is concluded that setting up at room temperature and pressure will not give significant errors until mine depths are greater than 1000 m. However, the effect on the rotameter reading is enough to take the float off scale at depths greater than 1500 m.

Recommended values for setting up the pump based on depth and an allowance for pressure drops on the loaded filter are given below.

Average depth below surface (m)	Airflow (L/min)	Motor speed (sec/indicated min)
0 - 300	2.05-2.15	59 -60
300 - 1000	2.1 -2.2	58.5-59.5
1000 - 2000	2.15-2.25	58 -59
2000 - 3000	2.2 -2.3	57 -58
Nominal values	2.0	60

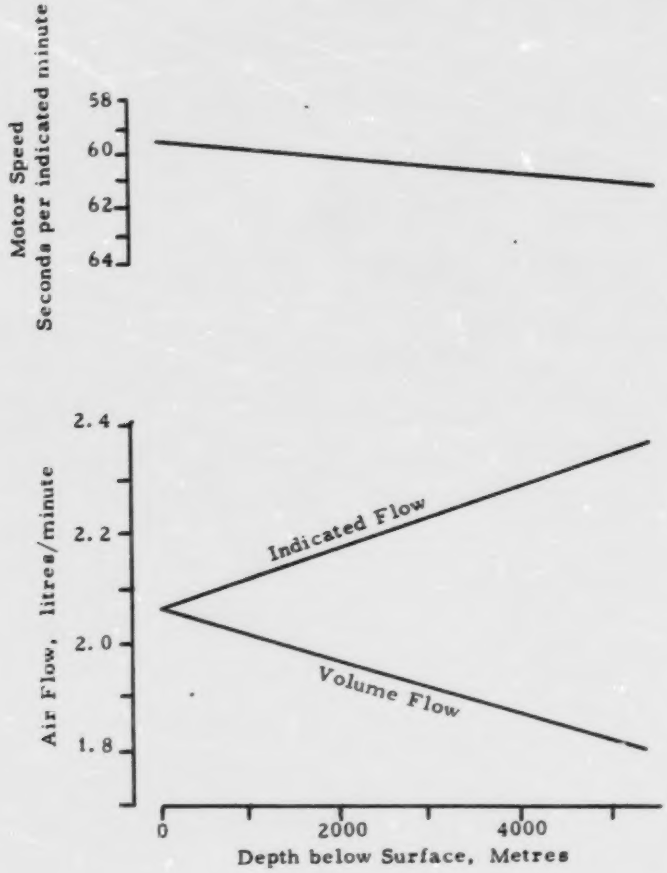


Fig. G.5 - Effect of mine depth on pump characteristics

G.3. Underground tests

Five CAMPEDS units were built for the Elliot Lake Laboratory and tested in one of the local mines. The samplers were worn by a representative section of the underground staff and miners. Careful measurements of airflow and motor speed were made both with the clean filter installed and after collecting the sample.

The first set of tests started with supervisors and progressed to the miners after a few days. The mine ventilation department arranged the issue of dust samplers and laboratory staff changed the cassettes and checked the instruments daily. A total of 86 samples were taken in 18 days. Sample losses occurred due to both mechanical faults and human error.

The results obtained in the trials are shown in Tables G.2 to G.6 where the mass of respirable dust collected or net weight flow rate before and after sampling, motor speed before and after sampling expressed as seconds per one-minute indication on dial, indicated running time in minutes, and clock running time in minutes for each instrument are given.

It can be seen that the samples averaged 0.5 mg in weight with a maximum of 1.1 mg and that on the average little change in airflow or motor speed occurred between measurements made before and after the sampling period. However, it was noticed that cold pumps indicated high airflow and presumably airflows after sampling were biased on the high side because the pumps

Table G.2 - Tests on dust sampler No. 1

Filter no.	Net wt	Flow rate L/min		Motor speed Sec/ind. min		Running time, min		
		Before	After	Before	After	Indicated	Clock	Diff, (I-C)
1	0.23	2.1	2.1	58.4	58.4	450	450	0
6	0.35	2.1	2.1	58.4	58.2	437	435	2
11	0.30	2.05	2.08	58.0	58.2	547	525	22
16	0.86	2.1	2.08	58.0	58.0	238	225	13
21	0.46	2.1	2.1	58.0	58.0	294	285	9
26	0.46	2.1	2.1	58.0	58.0	232	220	12
31	0.62	2.13	2.1	58.0	58.0	230	225	7
36	0.53	2.1	2.1	58.4	58.6)	955	420	40
41	0.64	2.1	2.1	58.4	58.4)		495	
46	0.48	2.1	2.0	58.2	58.0	528	510	18
51	0.34	2.1	2.15	57.2	58.0	510	490	20
56	0.52	2.1	2.1	57.6	57.6	525	515	10
61	0.19	2.14	2.15	59.4	59.4	525	518	5
65	0.62	2.12	2.2+	58.4	60.2	467	460	7
70		2.1		59.2	)	1008	1010	-2
70	0.55		2.11		58.8)			
71	0.44	2.1	2.1	58.8	58.4	499	495	4
82		2.09	2.1	58.4	58.0	511	510	1
Mean	0.475	2.102	2.102	58.3	58.4			+9

Table G.3 - Tests on dust sampler No. 2

Filter no.	Net wt	Flow rate L/min		Motor speed Sec/ind. min		Running time, min		
		Before	After	Before	After	Indicated	Clock	Diff, (I-C)
2	0.16	2.1	2.1	58.6	58.8	271	360	-89
7	0.28	2.1	2.0	58.6	59.6	245	235	10
12	0.39	2.12	2.15	58.4	59.0	456	450	6
17	1.03	2.05	2.08	59.0	59.2	485	475	10
22	0.30	2.05	2.17	59.0	58.0	483	470	13
27	0.20	2.08	2.1	58.6	58.6	229	220	9
32	0.21	2.1	2.07	58.6	59.6	229	225	4
37	0.33	2.1		59.6	)	849	360	9
42	0.35		2.1		59.2)		580	
47	0.85	2.05	2.05	59.0	59.0	524	520	4
52	0.73	2.18	2.0	58.0	58.4	503	490	13
57	0.40	2.0	2.1	58.4	58.0	518	495	23
62	0.42	2.12	2.2+	58.4	46.4	610	510	100
66	0.75	2.1	2.13	58.8	60.0	440	460	-20
78	0.37	2.12	2.0	58.8	62.0	471	490	-19
83		2.1	2.1	58.8	59.2	516	510	6
Mean	0.518	2.095	2.08	58.75	59.2			5

Table G.4 - Tests on dust sampler No. 3

Filter no.	Net wt	Flow rate L/min		Motor speed Sec/ind. min		Running time, min		
		Before	After	Before	After	Indicated	Clock	Diff, (I-C)
3	0.32	2.1	2.0	58.2	58.4	544	505	39
8	0.15	2.13	2.13	59.4	59.6	251	240	11
13	0.52	2.1	1.9	59.2	59.6	455	450	5
18	0.95	2.1	1.88	59.6	59.6	495	480	15
23	0.33	2.08	2.08	59.4	59.6	290	290	0
28	0.22	2.08	2.02	59.6	59.6	229	220	9
33	0.34	2.05	1.95	59.6	59.6	472	465	7
38	0.28	2.03		59.6	)	526	225	-69
43	0.21		2.1		59.2)		370	
48	0.59	2.1	2.05	59.2	58.4	528	515	13
53	0.38	2.1	2.05	59.4	59.4	461	470	-9
69	0.47	2.08	2.16	59.0	58.8	495	507	-12
72	0.66	2.09	2.1	59.0	58.0	509	497	12
75	0.41	2.08	2.09	58.0	59.0	515	497	18
79	0.52	2.1	2.1	59.0	57.6	521	502	19
84		2.06	2.04	59.0	58.8	529	513	16
Mean	0.423	2.085	2.04	59.15	59.0			10

Table G.5 - Tests on dust sampler No. 4

Filter no.	Net wt	Flow rate L/min		Motor speed Sec/ind. min		Running time, min		
		Before	After	Before	After	Indicated	Clock	Diff, (I-C)
4	0.46	2.1	2.1	58.4	58.0	275	255	20
9	0.17	2.08	2.0	58.0	59.2	137	235	-98
14	0.19	2.08	2.1	58.0	59.0	138	450	-312
19	0.59	2.1	2.13	58.4	58.0	85	470	-385
24	0.39	2.08	2.08	58.4	58.0	75	480	-405
34	0.24	2.1	2.2+	58.0	58.0	475	465	10
39	0.84	2.13	2.05	58.4	58.4)	694	165	3
44	0.91	2.1	2.1	59.0	58.4)		492	
49	0.50	2.1	2.1	58.2	58.0	542	520	22
54	0.47	2.1	2.18	57.6	56.4	517	515	2
59	0.42	2.1	2.18	55.6	56.0	538	515	23
67	0.52	2.1	2.1	58.0	58.0	492	485	7
73	0.41	2.09	2.12	60.0	57.2	528	516	12
76	0.92	2.1	2.05	58.6	60.0	498	495	3
80	0.49	2.1	2.02	60.0	60.0	489	495	-6
85		2.1	1.84	58.4	60.0	479	473	6
Mean	0.515	2.075	2.085	58.3	58.2			13

Table G.6 - Tests on dust sampler No. 5

Filter no.	Net wt	Flow rate L/min		Motor speed Sec/ind. min		Running time, min		
		Before	After	Before	After	Indicated	Clock	Diff, (I-C)
5	0.74	2.1	2.1	58.4	58.8	278	275	3
10	0.35	2.12	2.2+	58.4	58.4	360	345	15
15	0.64	2.1	2.1	58.4	59.6	456	450	6
20	0.30	2.13	2.13	58.6	58.6	236	225	11
25	0.50	2.13	2.1	58.2	59.2	445	405	40
30	0.38	2.14	2.1	59.0	59.0	230	220	10
35	0.23	2.07	2.05	59.0	59.2	228	225	3
40	0.42	2.1	2.2	58.4	56.4)	954	420	34
45	0.58	2.1	2.1	58.4	58.4)		500	
50	0.66	2.13	2.1	58.2	59.0	517	505	12
55	0.38	2.1	2.1	59.0	58.8	473	500	-27
60	1.10	2.08	2.1	59.2	57.0	516	495	21
64	0.41	2.11	2.1	58.8	59.2	527	--	
68	0.43	2.1	2.14	59.6	58.8	526	513	13
74	0.43	2.1	2.1	58.4	58.9	513	505	8
77	0.25	2.11	2.03	59.2	60.6	248	243	5
81	0.31	2.03	2.1	60.0	59.0	505	493	12
86		2.1	2.1	59.2	59.0	496	485	11
Mean	0.476	2.103	2.103	58.8	58.75			10

had not always reached room temperature when the flow checks were made. Mostly, good agreement existed between running times by the instrument indicator and the clock except when the instrument was switched off or failed.

Mechanical and electrical problems in the first series of tests and their solutions are listed below.

1. It was difficult to achieve a seal between the O-ring on the cassette and the cassette cover, due partly to distortion of the unmachined tube from which the covers were made. These were machined, using a larger O-ring and the O-ring groove on the cassette was remachined.
2. Big end bearings on two pumps seized up occasionally. The bearings were cleaned and oiled.
3. The electronic speed control failed on instrument No. 2, leading to a high motor speed throughout most of one shift. A temporary repair was made using a 22-ohm fixed resistor and a 5-ohm 1-watt potentiometer in series.
4. Exit pipes on the cassettes leaked. These were glued in place with epoxy resin.
5. One pump case came off a battery underground. Adequate care must be taken in machining the batteries to fit the pump case.

On completing these modifications a further set of underground tests was carried out, with mine staff looking after all procedures, except quartz analysis. A total of 130 samples were taken with very few samples lost due to human error or mechanical problems. The mine ventilation department staff was satisfied and ordered these samplers for routine use.

G.4. Pulsation Dampers

Work in the Elliot Lake Laboratory showed considerable differences in dust collection characteristics among three dust samplers from different manufacturers using the same size selector (3). Some of these differences are attributed to the effect of pulsating flow on dust collection in the cyclones used for size selection of respirable dust (3,11,12). The pulsating flow gives rise to greater deposition in the cyclone than a smooth flow and, therefore, to a lower estimate of the respirable dust concentration. The laboratory calibrations of cyclones reviewed by Lippmann were based, it is believed, on smooth flows (13).

The pulsating flow arises from using single-action diaphragm or piston pumps in the samplers and has a secondary effect on airflow calibration. Taper tube flowmeters are used as secondary standards and, if the float does not follow the pulsations, the root mean square of the airflow is read rather than the average, i.e., the flowmeter will read high, up to twice the average flow. Pulsation dampers have been used to give a reasonably accurate reading on a taper tube flowmeter calibrated on a smooth airflow (14).

This section describes studies on pulsation dampers for the modified Casella sampler combined with the cap lamp battery, as well as on some dampers built for the existing Casella pumps after Higgins and Dewell (2). Similar work on the MSA personal respirable dust sampler has been carried out in this laboratory.

The pulsation damper supplied with the Casella personal gravimetric sampler is a bulky separate component which can be awkward to use in some mining locations. It has also been found that frequent replacement of the rubber finger cotts is required and that they are prone to leakage.

Combining the personal gravimetric dust sampler with the cap lamp battery involves fitting the pump and pulsation damper into a minimum-sized holder above the battery. The layout of the unit was described earlier (G.1). Following the USBM study on symmetrical two-chamber pulsation dampers (15), it was decided to build a symmetrical four-chamber unit to fit the space available.

This unit was found to control dust collection adequately but too much error remained in the flowmeter reading. Some experimental units were therefore built to attach directly to the case of the original Casella personal sampler pump so that some of the variables could be investigated with less restriction on overall size (1). The parameters of the various dampers and the results of tests described in the two following sections are given in Tables G.7 and G.8.

Following this, a modified three-chamber version was prepared for the unit combined with the cap lamp battery and was tested.

G.4.1. Effect of pulsation dampers on dust collection

Tests on various pulsation dampers placed between a Casella personal sampling pump and its cyclone filter unit were carried out in an approximately 50% respirable coal dust cloud in the laboratory chamber. This dust cloud was chosen because it had shown the largest difference between size-selective dust samplers (3). The dust collected on each filter was compared with that collected by the large volume — 50 L/min — Hexhlett respirable dust sampler as this could be weighed with negligible error. Tests were also carried out with the same cyclone filter unit and a smooth airflow obtained from a vacuum pump and a critical orifice. The results are shown in Table G.7. It should be noted that the combination of the units — pump, pulsation damper, and filter-cyclone — were altered for each run so as to average out variations between the individual pumps and filter-cyclones.

It can be seen that the absence of a pulsation damper on this sampler leads to a low estimate of respirable dust (Code 1). The Casella (Code 2) and the laboratory-built damper with thin rubber (Code 5) are adequate to keep the dust collection within 5% of that with a smooth flow, while the laboratory damper with thick rubber (Code 4) and the single chamber proprietary damper (Code 6) do not. It should be noted that at the sampler's recommended airflow of 1.9 L/min the estimate of respirable dust is 10 to 15% higher than that given by the Hexhlett.

Table G.7 - Effect of pulsation dampers on dust collection with the Casella personal gravimetric dust sampler

Code	Pulsation damper			Dust collection expressed as fraction of that by Hexhlett	
	No. of chambers	Volume cm^3	Rubber thickness mm	Individual runs	Mean
1	None			0.43, 0.68, 0.61, 0.55, 0.78	0.65
2	2	32	0.18	1.04, 1.12, 1.20, 1.06, 1.18, 1.04	1.11
3	3	17	0.48*	1.04, 1.21, 1.12, 1.07, 1.11, 1.06	1.10
4	2	16	0.71	0.98, 0.96, 1.03	0.99
5	2	16	0.48	1.06, 1.18, 1.08, 1.10, 1.13, 1.03	1.10
6	1	9	0.35	1.15, 1.06, 1.04, 1.12, 1.06, 1.03	1.07
7	* Smooth flow			1.07, 1.13, 1.21, 1.11, 1.18, 1.21	1.15

*Final design of smoother for the prototype Casella personal samplers combined with the cap lamp battery.

Table G.8 - Effect of pulsation dampers on flowmeter reading Casella personal sampler pump

Test	Pulsation damper				Air flow		
	Number of chambers	Total volume cm^3	Total rubber area cm^2	Rubber thickness mm	Rotameter[1] L/min	WTGM[2] L/min	Excess reading R - WTGM L/min
1		none			3.4	2.00	1.4
2		smooth flow			2.02	2.02	0.00
3	2	16.0, 16.0	50, 50	0.18[3]	1.86	1.85	0.01
4	1	10.6	22	0.71	2.07	1.93	0.14
5	2	21.2	22	0.71	1.88	1.86	0.02
6	2	5.3, 5.3	11, 11	0.71	2.02	1.91	0.11
7	2	5.3, 5.3	11, 11	0.48	1.96	1.93	0.03
8	1	10.6	22	0.18[6]	1.99	1.96	0.03
9	2	5.3, 5.3,	11, 11	0.18	1.76	1.74	0.02
10	3	5.3, 2.0, 2.0	11, 4 4	0.18	1.94	1.93	0.01
11	4	2.0, 2.0, 2.0, 2.0	4, 4, 4, 4	0.18	2.04	1.89	0.15
12	2	2.0, 9.0,	0, 18	0.35	2.06	2.00[4]	0.06
13	1	9.0	18	0.35	1.89	1.83[4]	0.06
14	3	12.8, 2.2, 2.2	11.5, 4.6, 4.6	0.28	2.02	1.86	0.16
15	3	12.8, 2.2, 2.2		0.48[5]	1.91	1.89	0.02
16	3	12.8, 2.2, 2.2		0.18[6]	1.94	1.93	0.01

[1] Rotameter calibration adjusted to agree with wet test gas meter (WTGM) on smooth flow.

[2] WTGM.

[3] Supplied with Casella personal gravimetric sampler.

[4] Difference in volume between the two tests is presumably due to difference in length of tubing attached to pump inlet.

[5] Configuration chosen for modified Casella fitted to cap lamp battery.

[6] This rubber has less strength than others used.

G.4.2. Effect of pulsation dampers on reading of taper tube flowmeter

In this study the wet test gas meter, taper tube flowmeter, pulsation damper, and Casella pump were set up in series. The calibration of the flowmeter was adjusted to give the same reading as the wet test gas meter (WTGM), in series, using a smooth airflow from a critical orifice and vacuum pump. Results are given in Table G.8.

Note, the seventh column shows the excess reading on the rotameter due to pulsations in the airflow. In tests 3, 5, 7, 8, 9, 10, 11, 12, 13, 15, and 16 the error in flow rate reading, column 7, is less than 5% and in tests 5, 9, 10, 15 and 16 it is less than 2%.

G.4.3. Discussion on pulsation dampers

The experiments carried out and reported in Tables G.7 and G.8 as well as other less controlled experiments and observations suggest that the following factors should be considered in design and use of pulsation dampers:

(i) Rubber should be as thin as possible.

(ii) Volume and rubber area of the chamber nearest the pump inlet should be as large as possible; for the Casella pump tested, 5 cm^3 volume and 10 cm^2 rubber area appear to be the minimum acceptable.

(iii) There appear to be advantages in splitting up the pulsation damper into sections with an orifice between them as small as possible without too great a pressure drop; holes 2.5 mm in diameter were found to be a reasonable compromise.

(iv) Passage between the pulsation damper and pump should be as short and as large in cross section as practicable.

(v) Performance under suction is improved by using a support such as a coil spring under the rubber diaphragm to lessen volume decrease; presumable the spring-rubber combination is more elastic than thicker rubber of equivalent strength.

(vi) One manufacturer claims a dynamic action in the pulsation damper with tuning of spring, rubber diaphragm, and pump speed.

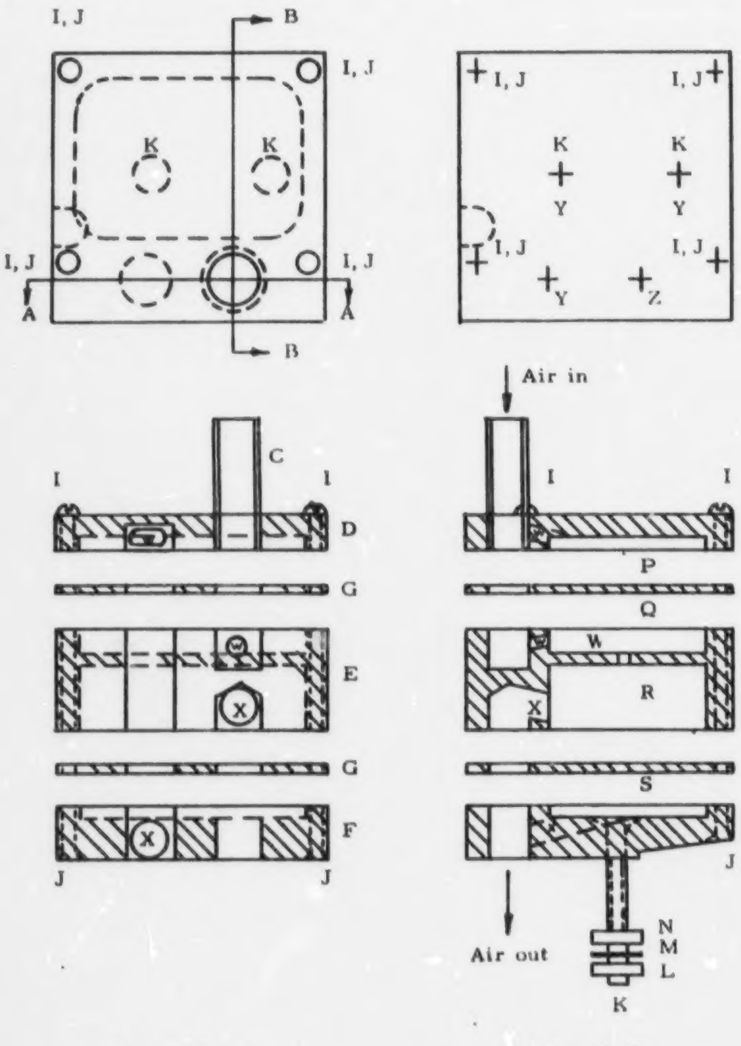
Clearly, both accuracy of dust collection and airflow are important in respirable dust samplers using cyclone size selectors and it is shown that the pulsations in airflow arising from the use of simple single-action air pumps can lead to errors in both these factors. These pulsations and their effects can be controlled by dampers.

Variations in dust collection of these samplers under set conditions in the laboratory are about ±5%, while in the field they may be ±10% or more. Reasonable accuracy is possible if the errors due to airflow pulsations can be kept within 5% for both dust collection and taper tube flowmeter reading. However, the requirement for dust collection at 5% is more stringent for cyclone size selectors and corresponds to a flowmeter reading change of about 2%.

The possibility of decreased effectiveness of the pulsation dampers at high pressure-drops, due to the decreased internal volume of the dampers from stretching of the rubber diaphragms may require a higher efficiency in the clean filter low pressure drop tests.

Inaccuracies in the use of a flowmeter due to pulsations can be overcome by calibrating against a wet test gas meter, spirometer or some other standard, but taper tube flowmeters or rotameters are in widespread use as secondary airflow standards and are the only instruments available in many laboratories. Thus, flow should be smooth enough on all sampling instruments for rotameters to give a sufficiently accurate assessment of flow rate.

Pulsation damper developed for Casella model B personal dust sampler are shown in Fig. G.6 and G.7. The design for the Casella B was elaborate because it was mounted outside the pump case and the rubber diaphragms must be protected from grit.



Section A-A

Section B-B

PARTS LIST

C - Tube 5/16" O.D. x 3/16" I.D. x 1" Long.
D - Plate 2" x 2" x 1/4"
E - Plate 2" x 2" x 5/8" or 3/4", tapped to receive screw I and J.
F - Plate 2" x 2" x 3/8".

Material: light alloy
All mating surfaces to be flat

G - Rubber, 2 pieces 2 1/2" x 2 1/2" x 0.025"
These are stuck to piece E
Holes are cut with hot soldering iron
Edges trimmed after assembly.
H - "O" ring 5/16" x 7/16" x 1/16" (set to seal on pump inlet tube)
I - 4 screws RHD 1/8" O.D. x 1/2" long.
J - 4 screws CSK 1/8" O.D. x 1/2" long.
K - 2 screws CSK 1/8" O.D. x 3/4" long.
L - 2 nuts to fit K.
M - 2 lock washers to fit K.
N - Clamping plate 1.25" x 0.25" x 0.125" with 2 clearing holes to fit K.
O - Milled relief 0.25" x 0.25" x 0.125" deep in part F to clear screw on pump case.

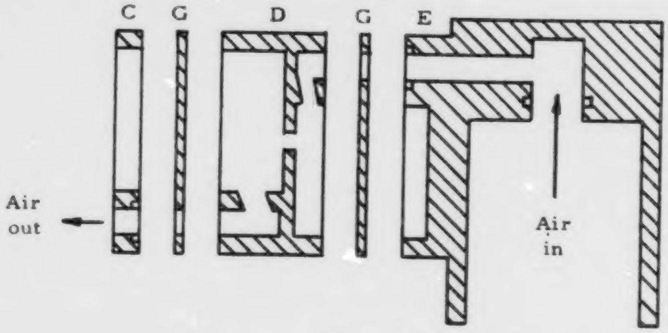
Milled recess in plates D, E and F, 1.625" x 1.16" x 0.25" - depth as shown in B-B.
P - 0.1"
Q - 0.187"
R - 0.375" if E 5/8" thick, and 0.50" if E is 3/4" thick.
S - 0.1"

Milled relief for opening pump case lid.
T - 0.70"
U - 0.13"
V - 0.07"

Air passages
W - 0.125" dia.
X - 0.3" x 0.125" or equal area.

Attaching to pump case
Y - Matching holes in pump case.
Z - Pushes on pump inlet tube. Check size of tube for O ring 'H' seal. Remove casting burr if necessary.

Fig. G.6 - Pulsation damper for Casella Model B personal dust sampler


C
G
D
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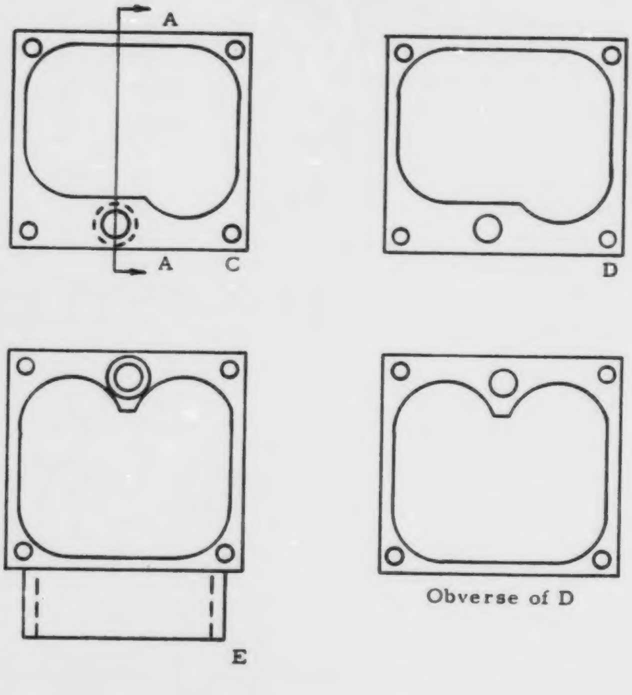
E
Air
out
Air
in

Section A-A



Obverse of D

Fig. G.7 - Pulsation damper and cyclone-filter holder for mounting on cap lamp battery.

G.5. References

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APPENDIX H

ANALYSIS OF ERRORS IN GRAVIMETRIC AND X-RAY DIFFRACTION ASSESSMENTS

Random and systematic errors can occur in gravimetric and X-ray diffraction assessments of dust samples. This Appendix describes an experimental assessment of errors. Those detected were mainly random, the only systematic error detected was in calibrating the X-ray diffraction equipment for some samples not taken by the normal method. Types of errors detected were:

1. weighing errors due to balance and interpolation of 5th place in reading the optical scale
2. damage in handling:
   - (a) thin slivers of filter material from the cutting operation are often left on the filters and may be lost after taring if they are not removed
   - (b) other damage resulting in loss of filter material
   - (c) gain of extraneous material from dirty filter holder
   - (d) transfer of coarse non-respirable dust from cyclone by either rough handling or horseplay
3. human error — substantial transcription and arithmetical errors have been detected
4. ashing — the observed changes in weight of clean filters when ashed are close to those in 1 above; however, overheating can change the filter weight and careless handling can lead to loss in ash or interchanged filters, with subsequent confusion
5. errors in X-ray assessment.

H.1. Weighing Errors

Since all filters are weighed two and possibly three times, it is important to know the limits of accuracy of the balance. Systematic errors are of little concern here since only a weight difference is needed and the same balance is used for all weighings.

A semi-micro Mettler balance with scale divisions of 0.05 mg, read to 0.01 mg is used for most work. When greater precision is needed a microbalance is used. Random errors in filter weights occur due to variations both in the balance and in fifth-place reading.

To determine the magnitudes of the random errors, ten clean silver membrane filters were selected for repeated weighings. They were read after equal time intervals for one month and the changes in weight readings were determined. The results are given in Table H.1 and a frequency distribution is shown in Fig. H.1. When the subjective nature of estimating the fifth decimal place with possible preferences is taken into consideration, the error curve can be considered to be Gaussian. This method of analysis could also tend to make the peak higher and narrower than it really is. The most probable error is zero and no errors greater than ±80 μg were observed. The standard deviation, σ of the results is ±30 μg taken to be the accuracy of weighing and fifth place reading errors.

H.2. Errors in Gravimetric Assessment

A large proportion of the field samples indicating over 100% quartz were low in weight and it seemed likely that errors in gravimetric assessment of total respirable dust could be the main cause of the high quartz indication. Thus, an estimate of the errors in the mass of total respirable dust was made, assuming:

1. X-ray analysis of quartz is accurate
2. quartz content expressed as a percentage is constant in the mineral dust in any one mine
3. proportion of non-mineral dust in the samples showing unduly high quartz percentage is negligible.

For each mine an average quartz content, Q%, was determined from ashed heavy samples of more than 1 mg, and this was used to determine the expected weight of the dust sample on all low weight samples showing higher than expected quartz percentages.

Table H.1 - Distribution of random errors in weighing

Filter no.	Mean weight (mg)	Frequency of deviation from mean for each value, in mg														
		-.07	-.06	-.05	-.04	-.03	-.02	-.01	0	-.01	.02	.03	.04	.05	.06	.07
W 1	157.15	0	0	0	0	1	1	0	4	0	1	1	0	0	0	0
W 2	148.32	0	0	1	0	2	0	0	1	2	0	1	0	1	0	0
W 3	179.65	0	0	1	0	1	1	1	1	0	1	1	1	0	0	0
W 4	161.51	1	1	0	0	1	0	0	2	0	0	1	0	1	1	0
W 5	179.90	0	0	1	0	1	0	2	0	2	0	2	0	0	0	0
W 6	171.91	0	0	0	1	0	3	0	0	0	2	0	2	0	0	0
W 7	179.00	0	0	0	1	0	2	0	1	2	2	0	0	0	0	0
W 8	171.40	0	0	0	0	0	0	2	3	3	0	0	0	0	0	0
W 9	176.52	0	0	0	1	0	0	2	2	3	0	0	0	0	0	0
W10	176.01	0	0	0	1	0	0	4	1	0	0	0	2	0	0	0
Totals		1	1	3	4	6	7	11	15	12	6	6	5	2	1	0

$\sigma = \pm 0.03$ mg

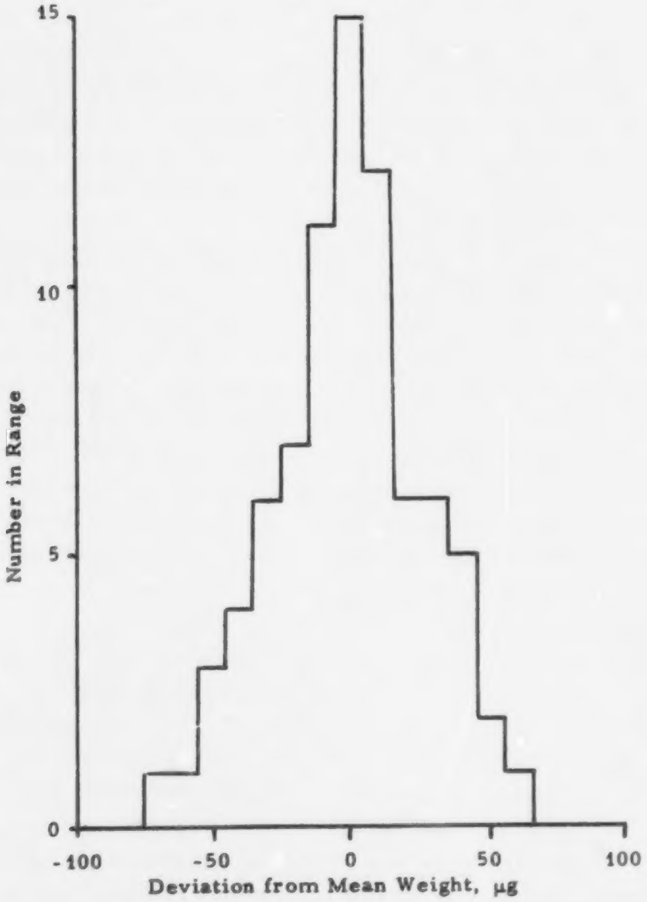

Fig. H.1 - Distribution of errors associated with weighing

$$W_E = 100 \cdot \frac{Q}{Q\%}$$

where Q is the XRD assessment of quartz, and Q% is the appropriate expected quartz percentage for that mine. An estimated error, ε, is then determined as:

$$\varepsilon = W_M - W_E$$

where W_M is the measured weight of dust.

This procedure determines only those samples which are lighter than expected and thus only the errors with negative values are detected.

The distribution curve of the estimated errors is shown in Fig. H.2. Figure H.3 shows correlation between the estimated error and the expected weight of mineral in the sample, plotted separately for mines with high and low quartz contents. The technique suggests that substantial errors can occur in gravimetric assessment, but it should be noted that method of selecting samples may well omit many samples with small errors and the proportion of large errors is thus likely to be overestimated.

Figure H.4 compares the cumulative probability curve of the estimated errors in gravimetric assessment with weighing errors. The estimated error curves suggest three error terms of substantially different magnitudes:

1. a standard deviation of about 35 μg on all samples
2. a standard deviation of about 80 μg affecting about one third of the samples
3. an error term greater than 100 μg affecting less than 5% of the samples.

The first error term is very close to errors, σ = 30 μg, associated with the balance and fifth-place reading errors.

Identification of the origin of the second error term is difficult but could include handling and transcription errors in fourth-place readings.

The third error term is substantial but fortunately only affects less than 5% of the samples. This portion of the curve does not contain sufficient points to be accurate.

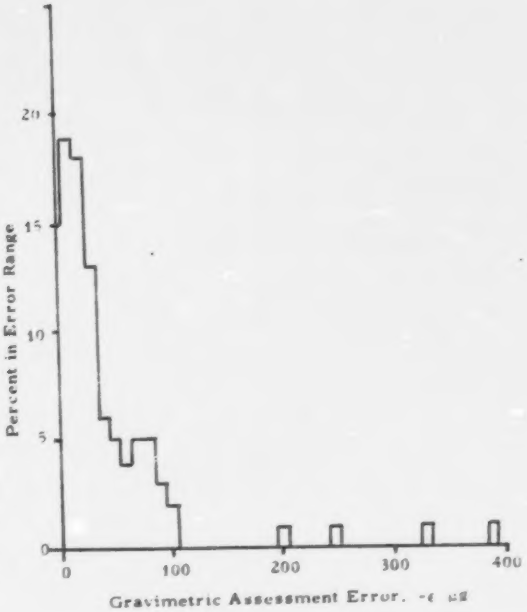

Fig. H.2 - Distribution of negative errors in gravimetric assessment calculated from samples showing high quartz contents

The presence of combustible dust would lead to an increased magnitude of the expected error equal to the mass of combustible dust. On all ashed samples analyzed to date the combustible portion has formed about 50% of the total respirable dust. Thus, the additional expected error due to ignoring combustible dust could be as great as the expected mineral weight. The average for 95% of the samples showing errors of less than 0.09 mg was 0.18 mg. However, because these samples were selected as showing higher than expected quartz contents they probably average lower in combustible dust. A reasonable description of the errors in gravimetric assessment of total respirable dust is that 90% of the samples have gravimetric assessment errors less than 100 μg in magnitude, and over 97% have errors less than 150 μg. The smaller errors are probably evenly distributed on either side of

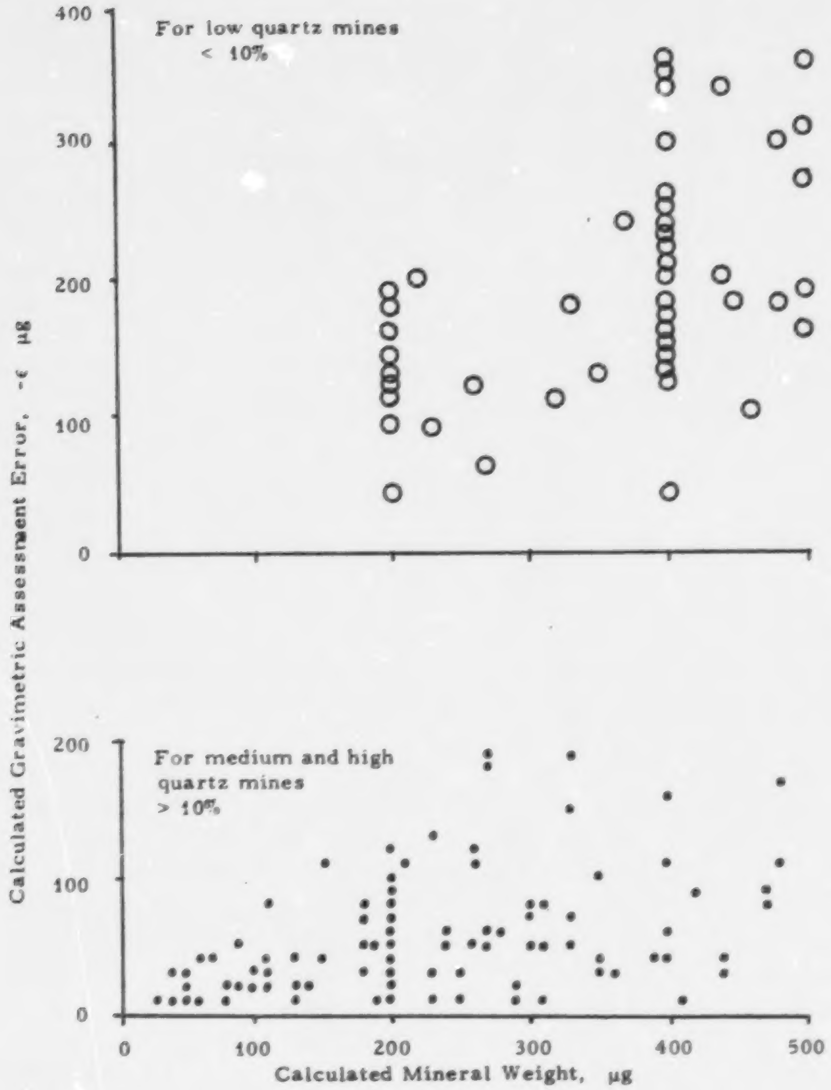

Fig. H.3 - Gravimetric assessment error plotted against calculated mineral weight

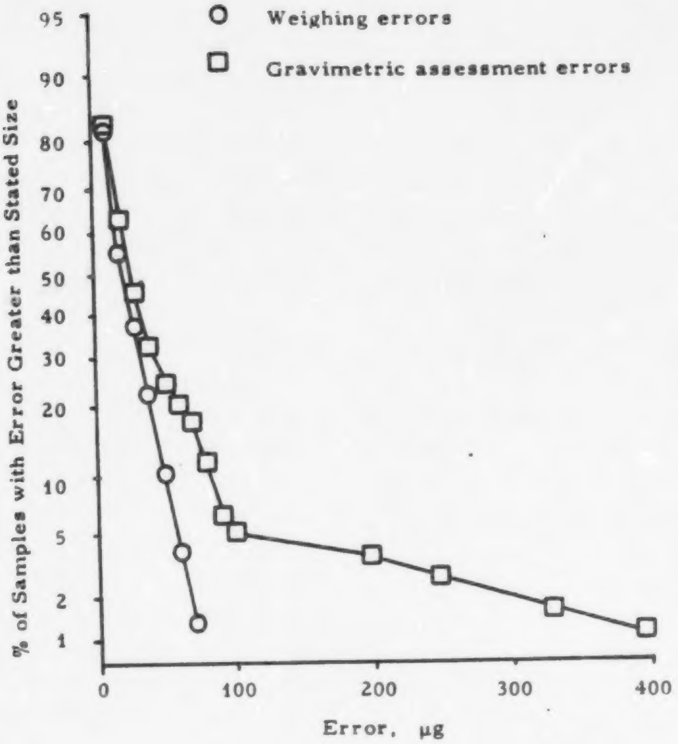

Fig. H.4 - Cumulative probability curves of weighing and gravimetric assessment errors

zero, and errors over 0.15 mg may tend to be more negative than positive if the major cause is filter damage.

As these estimated errors in gravimetric assessment are in reasonable agreement with experience, apparently gravimetric errors can give substantial errors in calculated quartz percentages on individual samples. It is concluded that useful figures for quartz percentages can only be obtained on heavy samples or as the mean of a number of samples.

H.3. Errors in X-ray Assessment

A number of errors can occur in X-ray assessment as discussed previously. In this section relevant experimental results are presented and given in two categories: measurements on standard samples, and duplicate measurements on samples.

Measurements on standard samples

The use of standard quartz samples was introduced to overcome secular variations in X-ray output and in adjustment of the equipment. X-ray intensity from standard samples dropped 50% after installing of a new chromium tube during a target 6-month period and running time of 2200 hours. This is a very significant reduction in X-ray tube output and thus must be included in any assessment of repeatability of measurement. However, running times have not been recorded and the only time variable that can be used is the number of batches or samples run. As the number of batches is easier to check, this has been used in a linear regression analysis of the relationship between batch number and the measured peak area of one of the two standards used. The regression analysis was done on the two most-used diffraction lines, and results are given in Table H.2.

Table H.2 shows that the average change in X-ray intensity per batch of samples is negligible at about 0.3% and that the standard error of the measurement is 7.5% when corrections are made for decrease in intensity with time (batch number). However, other sources of error in the intensity which apply to both the samples and the standards are:

1. slight variations in setting of the X-ray diffraction equipment
2. changes in X-ray intensity which are not linear with batch number.

Thus, the actual variation from sample to sample because of variation in measuring standards is less than the 7.5% given above and is probably less than 3%.

Duplicate Measurements

The first set of duplicate measurements was undertaken when selecting 125 samples for an interlaboratory check. These covered a wide range of mines and most had been analyzed two years or more previously. The analyses are expressed as best estimates. Distribution of the differences between the two analyses was approximately Gaussian centered on a zero mean difference. Figure H.5 shows the distribution plotted against mass. It can be seen that the maximum differences range between 30 μg on light samples and 60 μg on heavier samples.

Between these two sets of measurements improvements in technique and equipment were introduced. The samples had been stored possibly damaged leading to loss of quartz or had been contaminated leading to gains in quartz; thus, differences between the two analyses might not be representative of the analyses now undertaken.

The second set of duplicate measurements was made on 33 carefully selected undamaged samples, which were analyzed twice within a 4-week period. Results are given in Fig. H.5, and show that the differences between the two analyses range from 10 μg on light samples to 3% on heavy samples, an improvement over the first set, and probably showing the best repeatability available. However, damage and interference are probably the major factors in limiting accuracy of quartz analysis.

Table H.2 - Statistical linear regression analysis of X-ray intensity (peak area) from one quartz standard

	X-ray diffraction line	
	1,0,1	1,1,2
Intercept (≡ intensity using new tube)	25180	3761
Regression coefficient	-65	-8.5
Change in intensity per batch[1], %	-0.35	-0.3
Correlation coefficient	-0.954	-0.948
Standard error of estimate	1213	215
Reliability of measurement[2], %	7.5%	7.5%

1 Regression coefficient divided by the mean value of the X-ray intensity · 100.

2 Standard error divided by the mean value of the X-ray intensity · 100.

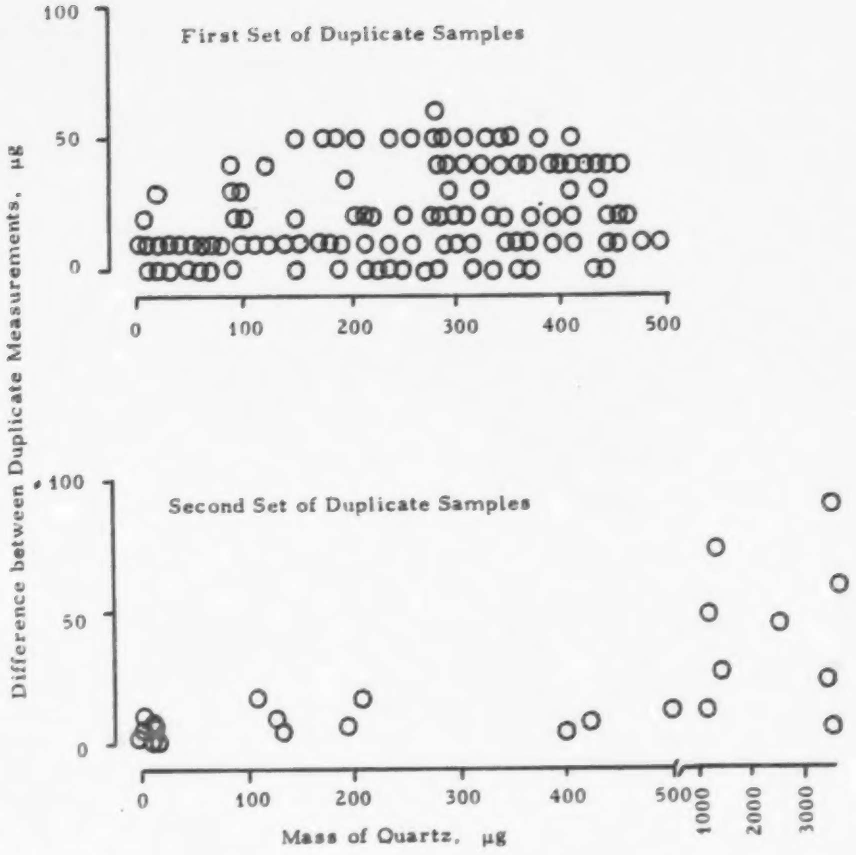

Fig. H.5 - Differences between duplicate measurements of quartz

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Section 1—Chapter 8

CAMPEDS—A Dust Sampling and Assessment System for Use in Hard Rock Mines

by

G. Knight

Annals of Occupational Hygiene 30(2) 139-151 (1986)

CAMPEDS - UN ÉCHANTILLONNEUR DE POUSSIÈRE ET UN SYSTÈME D'ÉVALUATION DESTINÉ À ÊTRE UTILISÉ DANS LES MINES DE ROCHE DURE

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Résumé

Un échantillonneur de poussière permettant une évaluation gravimétrique par pesée et une évaluation de la concentration de quartz par diffraction des rayons X, mis au point par le laboratoire d'Elliot Lake pour être utilisé dans les mines de roche dure, est décrit. Les compromis entre les exigences parfois contraires de l'échantillonnage, de l'incinération, de la pesée, de l'évaluation de la concentration de quartz et du fonctionnement de la pompe sont soulignés. On insiste sur la nécessité de définir le système d'échantillonnage et d'analyse de la poussière totale, avec les méthodes d'étalonnage.

Le système comporte : (1) un petit classificateur à chocs et un porte-filtre cassette intégral, montés avec la pompe sur la batterie de la lampe de chapeau du mineur; (2) un filtre à membrane d'argent de 25 mm de diamètre; (3) une évaluation gravimétrique avant et après l'incinération; (4) une évaluation par diffraction des rayons X de la concentration de quartz; (5) un diffractomètre commandé par ordinateur. Il existe d'autres versions pour l'échantillonnage avec l'appareil porté au revers ou, si l'on utilise une pompe branchée sur une batterie légère, pour l'échantillonnage individuel ou en station fixe sur une surface. Le classificateur est étalonné par comparaison avec un échantillonneur de référence. Le dispositif de détermination de la concentration de quartz est étalonné à partir d'échantillons recueillis dans un nuage de poussière atmosphérique de quartz pur.

Les avantages du système sont les suivants : détermination rapide de la concentration de quartz qui ne demande pas beaucoup de temps et d'habileté de la part du technicien; grande capacité de débit (10 000 échantillons par an par diffractomètre X); logistique simple pour l'échantillonnage individuel dans les mines souterraines; échantillonneur robuste et échantillons de bonne qualité; grande résistance à la contamination de la poussière inhalable par la poussière grossière.

Ann. occup. Hyg., Vol. 30, No. 2, pp. 139–151, 1986.
Printed in Great Britain.

0003-4878/86 $3.00 + 0.00
Pergamon Journals Ltd.
British Occupational Hygiene Society.

CAMPEDS—A DUST SAMPLING AND ASSESSMENT SYSTEM FOR USE IN HARD ROCK MINES

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Abstract—A dust sampling system comprising gravimetric assessment by weighing and quartz assessment by X-ray diffraction, as developed by the Elliot Lake Laboratory for use in hard rock mines, is described. The compromises made between the sometimes conflicting requirements of sampling, ashing, weighing, quartz assessment and pump performance are stressed. Emphasis is given to the need for defining the total dust sampling and analysis system, including the calibration procedures.

The system incorporates: (1) a small impactor size selector and integral cassette filter holder, mounted with the pump on the miner's cap lamp battery; (2) a silver membrane filter 25 mm in diameter; (3) gravimetric assessment before and after ashing; (4) X-ray diffraction assessment for quartz; (5) computer controlled diffractometer. Alternatives are available for lapel sampling or, using a pump driven by a light-weight battery, fixed station sampling or personal sampling on surface. Size-selection is calibrated, by comparison with a reference sampler, while quartz calibration is by collection of samples in a pure quartz airborne dust cloud.

The advantages of the system are: rapid assessment of quartz, with low requirements for technician time and skill; high throughput capacity—10 000 samples per year per X-ray diffractometer; easy logistics for personal sampling in underground mines; robust sampler and samples; high resistance to contamination of respirable dust by coarse dust.

INTRODUCTION

DUST SAMPLING and assessment in mines are concerned with estimating the health effects of dust and guiding the control measures. It is considered in many hard rock mines that some mineral components, such as free silica and rock fibre (including asbestos), are particularly hazardous to health. Of these, the free silica form, quartz, is widespread in many mines and requires major consideration.

The last few decades have seen the introduction of a substantial amount of diesel engine powered equipment in mines. From the airborne dust point of view, this is characterized by the emission from the exhaust of a very substantial quantity of fine, mainly combustible, particulate into the mine atmosphere. As yet, the exhaust particulates have an unknown effect on health (KNIGHT, 1980a; FRENCH and ASSOCIATES, 1978).

The use of compressed air powered equipment also introduces substantial quantities of combustible aerosol into the atmosphere (KNIGHT, 1980a). This is believed to be in the form of a fine mist, derived from lubricating oil, and collected on the filters of gravimetric samplers and weighed.

Size-selective respirable dust samplers are now widely used in coal and other mines and in surface industries; such samplers are accepted as a desirable element of dust sampling to estimate the health risks (ORENSTEIN, 1960; ISO, 1983). For the major hazard in mining, i.e. the pneumoconioses, the portion of dust reaching the alveolar region, usually termed the 'respirable dust', is the most important. It is usually considered that, if this fraction is measured and controlled, then the coarser fractions will not be a problem. There are possible exceptions, such as in the potash mines where dry cutting is used. Where this is the case, the airborne dust has a much higher than normal proportion of coarse particles, so the inhalable fraction, or that size fraction deposited in the tracheo-bronchial region, should be measured.

This report is concerned with the development of a sampling system with gravimetric assessment of the respirable dust and assessment of the quartz fraction by X-ray diffraction. This system is known as CAMPEDS—Canadian Mines Personal Dust Sampling System.

This paper is concerned with presenting the essential elements of the respirable dust sampling and assessment system. A more detailed account of the complete system and its development is available (KNIGHT, 1978) and papers on some aspects have been published (KNIGHT, 1980b, 1983; KNIGHT *et al.*, 1972, 1974a,b, 1980).

Preliminary investigations for work on the XRD assessment of one fibrous mineral has been carried out, but the determination of fibres in general requires very different techniques and it is not considered further here (ISO, 1984).

OUTLINE DESCRIPTION

The Canadian Mines Personal Dust Sampling System, CAMPEDS, is an attempt to meet the personal dust sampling requirements of hard rock underground mining with its emphasis on the control of free silica. The major objectives in addition to previous applications of gravimetric sampling are the improved ease of handling, use and personal fitting to the miner, as well as the minimization of laboratory costs of quartz assessment. The sampler and sampling head are shown in Figs 1 and 2.

The major features of the CAMPEDS system are:

(1) aerodynamic size selection matching the British Medical Research Council (BMRC) respirable dust specification (HAMILTON and WALTON, 1961);

(2) combination of the sampling head and pump with the the cap lamp battery to maximize ruggedness and acceptability to underground miners;

(3) a sampling head that compactly combines a two-stage impaction size selector

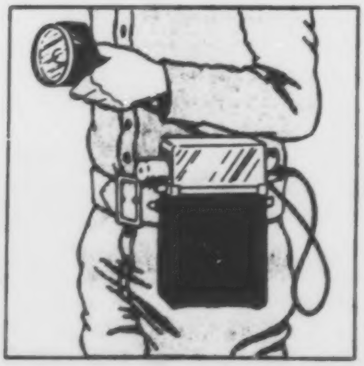

FIG. 1. The CAMPEDS dust sampler in use.

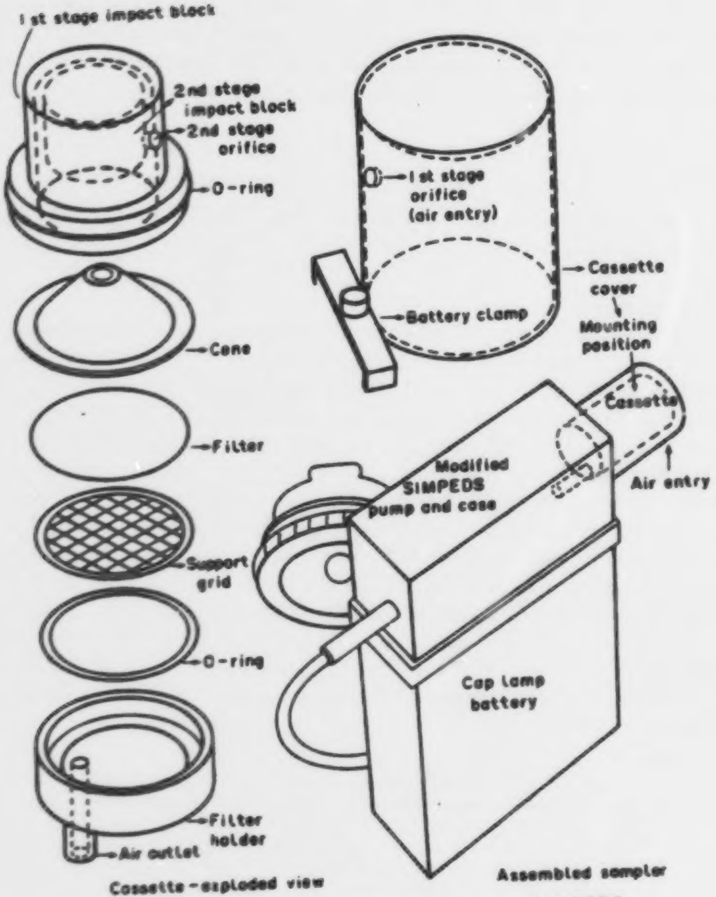
1st stage impact block
2nd stage impact block
2nd stage orifice
O-ring
Cone
Filter
Support grid
O-ring
Filter holder
Air outlet
Cassette - exploded view
1st stage orifice (air entry)
Cassette cover
Battery clamp
Mounting position
Cassette
Air entry
Modified SIMPEDS pump and case
Cap lamp battery
Assembled sampler

FIG. 2. Cassette filter holder and size selector for the CAMPEDS.

and a cassette filter holder for 25 mm diameter filters. Special precautions are taken to prevent transfer of coarse dust from its collection zone to the respirable dust filter. The flow rate is 2 l. min^{-1};

(4) direct-on-filter quartz assessment to reduce laboratory handling and costs;

(5) use of silver membrane filters;

(6) ashing—the silver membranes can be heated, *circa* 500°C, without change in weight;

(7) quartz assessment by quantitative X-ray diffraction;

(8) computer data reduction and control to minimize technician input, as well as assessment time and interference effects;

(9) calibration procedures—size selection. The orifice diameters and the flow rate

of the impaction size selector were adjusted until the CAMPEDS head agreed with the reference size selector, a Hexhlet (KNIGHT, 1978);

(10) calibration procedures—quartz assessment. Weighed samples collected using the CAMPEDS sampling head in a pure quartz dust cloud were used to calibrate the X-ray diffractometer. The physical reasons for the departures from linearity were identified and compromises made for running field samples.

DETAILED DESCRIPTION OF SOME ASPECTS

In this section some aspects of the CAMPEDS system are described together with their justification.

Aerodynamic size selection

Two size selection specifications for respirable dust samplers have been internationally accepted for use in the working place (ORENSTEIN, 1960; ISO, 1983; HAMILTON and WALTON, 1961; ACGIH, 1974). These lie within the range of human variability and can be expected to indicate dust concentrations differing by 20% in mineral dust clouds (KNIGHT and KIRK, 1982; MOSS and ETTINGER, 1970; LYNCH, 1970). On the basis that either of these is acceptable when used with a matching dust target level, the choice should be based on performance criteria in the specific task. Most size-selective samplers can be set to give a reasonable match to either the BMRC or the ACGIH specifications by choice of flow rate. With cyclone and impactor size selectors the mass of dust collected is almost independent of the flow rate, owing to changes in size selection compensating for the change in volume (TREAFTIS *et al.*, 1984; COCHRANE *et al.*, 1971). For a given device, the flow rate for the ACGIH specification is about 25% higher than for the BMRC. Thus, for this type of size selector, the choice can be based on the mass of dust collected and the airflows required to match each specification, as shown in Table 1.

TABLE 1. PERFORMANCE OF SIZE SELECTORS

Sampler	Relative respirable mass	Airflows L min^{-1} for selection characteristic BMRC	ACGIH
CAMPEDS impactor	1.05	2.0	2.4–2.5
10 mm nylon cyclone	0.7	1.33	1.7

For the same thickness of mineral dust collected for presentation to the X-ray beam, the ACGIH specification requires higher airflows. When diesel exhaust particulates are present, much higher resistances to airflow are found. Clearly, the BMRC specification is more suitable for direct-on-filter XRD assessment of quartz and other minerals in hard rock mines.

A second important factor is size-selection calibration procedures. The Elliot Lake Laboratory has used comparison with a 'reference' dust sampler in a polydisperse dust cloud rather than the 'monodisperse' dust cloud technique recommended by ACGIH (KNIGHT, 1978; LIPPMANN, 1970), because calibration is much quicker and less apparatus is required. A modified Hexhlet sampler to the BMRC specification has been used as the reference dust sampler (KNIGHT, 1978; KNIGHT and KIRK, 1982; COCHRANE *et al.*, 1971; WRIGHT, 1954; KNIGHT, 1984).

Sampling head

The sampling head (Fig. 2) consists of:

(1) a two-stage impaction size selector;
(2) two adhesive (petroleum jelly) coated coarse dust collection zones;
(3) a labyrinth seal (tortuous air path);
(4) a metal right cone to expand the air flow;
(5) a filter for collection of respirable dust;
(6) a 25-mm diameter filter;
(7) a backing plate;
(8) an elastomeric O-ring;
(9) casing components;
(10) a leaf spring;
(11) a 2 l. min^{-1} air flow.

The impaction size selector was chosen for its compactness (JACOBSON, 1973; TOMB and TREAFTIS, 1975) and expected ease of reproducible manufacture, as the main critical dimensions are the diameter of the drilled orifices. The two-stage impactor was introduced to increase the coarse dust holding capacity from 0.5 to 5 mg. The impaction size selector is very versatile and can, by choice of orifice numbers and sizes, be made to fit any flow rate and size-selection specification (MARPLE and MCCORMACK, 1983). However, our experience is that the theoretical performance is very easily modified by unexpected extra dust collection and experimental calibration of the complete sampling head is essential.

To date, a modified CAMPEDS sampling head has been used to collect both total and respirable dust in potash mines, where, because water cannot be used for dust suppression, the dust levels are high. A 4 l. min^{-1} air flow head has also been designed and is being tested and is expected to give higher dust and quartz assessment sensitivities and be usable with the improved personal sampling pumps and lower dust concentrations now found.

Items 2 and 3, adhesive coating and labyrinth seal, were introduced to further reduce the small proportion, about 1 in 20, of personal respirable dust samples which were contaminated by coarse dust when commercial cyclone size selectors and filter holders were used. Laboratory tests showed that about one fifth of the collected coarse dust could be transferred to the filter when the cyclone was turned upside down and gently tapped. The characteristic appearance of this effect was loose flocs of dust on top of, or partially embedded in, the compact layer of collected respirable dust.

Items 8 and 10, elastomeric O-ring and spring, ensured a good filter seal during sampling, together with easy filter handling. No dust deposits on the supposedly clean clamped edge have been seen, unlike our experience with samples received for quartz analysis from mines using 10 mm nylon cyclones and press fit plastic filter holders, when up to one third show such deposits (KNIGHT, 1985).

SCHMIDT and RAPPAPORT (1983) have measured edge leakage with the press fit filter holders.

Sampling pump

The SIMPEDS pump, manufactured by Casella, has been used in the version to fit the Oldham miner's cap lamp battery. The Wheat and MSA batteries used in Canada can be hand-filed to fit.

Tests on the pump at simulated depths in mines from +300 to −3000 m above sea level showed that:

(1) the flowmeters change calibration about 5% per 1000 m, as expected from the density change;

(2) the volume (at ambient conditions) of air pumped decreases with depth in the mine, i.e. with increasing air density.

The SIMPEDS pump could be set to operate correctly at depth by increasing the surface settings of flow rate by 3% and motor speed by 1% for every 1000 m of depth.

Tests on temperature at 0 and 20°C and surface air pressure showed small effects varying from pump to pump. It was noticed, however, that a cold pump brought into a warm room indicated a low airflow which recovered when the pump was warmed. This could possibly have been due to condensation on the filter in an excessive pressure drop.

Sampling position

The early tests showed sample and equipment losses due to difficulties in fitting shoulder harnesses to miners and catching equipment when climbing ladders, etc. The suggestion (Harris and Maguire, 1968) of combining the dust sampler with the cap lamp battery was considered. It was thought that, because of careless handling by miners, the mounting of the sampling head on the cap lamp would render the dust deposit prone to damage resulting in large errors in direct-on-filter assessment of quartz. This was overcome by mounting the sampling head directly on the belt (Fig. 1).

Tests (Knight, 1978) comparing two sampling heads on miners showed insignificant differences in dust levels between the belt and lapel positions, unless oilskins were worn as in roof bolt drilling with a stoper, when both positions were unsatisfactory. The miner could, however, be persuaded to wear the cap lamp battery with sampler and head outside the oilskin; samples were then probably reasonably representative of his dust exposure. There are some jobs in underground mining where a belt-mounted sampler could be expected to over-estimate the miner's exposure. These would include crusher operators and belt-pickers, where the dust source is consistently at a low level and at head level the air may be cleaner. In conclusion, it was considered that, while not meeting the usual requirement of being within the 300 mm diameter hemisphere centred on the nose, the belt mounting position is far more suitable for the rough conditions prevailing for many miners. Thus, this is the recommended sampling position. However, where lapel mounting is required this can be accommodated by use of an accessory kit. Brackets should be placed near the driver on a vehicle for the cap lamp battery and sampler both to bring the sampler into the airstream and to improve the driver's comfort.

The combination with the cap lamp battery could also be used to simplify the issuing and return of samplers, in that a tag hung at the man's normal lamp could tell him to take a special lamp number *x*. Automatic switching on while disconnected from the charger could be arranged, which, together with a timer, would reduce the demands on the dust sampling technician.

Use of the SIMQUADS pump (Casella Ltd) with a much lighter battery provides for both personal sampling on surface and fixed station-sampling anywhere.

Filter selection

A silver membrane filter 25 mm in diameter with a 5 μm pore size has been used as the best compromise between the sometimes conflicting requirements of cost, sampling-pump performance, thick dust layer and low background for the quartz peaks in X-ray diffraction.

Collection efficiency. Filters of pore size 0.8 μm are usually considered to be highly efficient dust collectors in air sampling. It was shown, using 0.8 μm back-up filters, that the penetration of respirable quartz dust through all types of 5 μm pore size filters examined was not detectable and they could, therefore, be considered efficient in sampling mineral dust.

Hygroscopic characteristics. Silver membranes are non-hygroscopic, as is desirable for sampling in humid atmospheres and for ease in weighing.

X-ray diffraction background. The silver membrane filters give a count almost as low as the empty sample holder in the regions of the four strongest quartz diffraction lines. The next most suitable filter materials, vinyl membrane and Nuclepore, have twice the background level at the strongest quartz peak and broad peaks at the second strongest quartz peak.

Handling. The silver membrane filters are robust and do not gather an electrostatic charge, unlike many of the plastic membrane filters.

Ashing. The silver membrane filters can be heated to about 500°C without a change in weight, thus permitting easy determination of the 'combustible', or its corollary 'ash', portions of the respirable dust. Blank filters should be tested, though, as some filters are contaminated before receipt and the process is sensitive to higher temperatures and sticking to the 'tables' used in the oven. Tables bent from thin 316 stainless steel sheet are used in a muffle furnace. Low temperature ashing cannot be used, as the blank filters increase in weight.

Filter size. A filter size of 25 mm diameter with a 20 mm diameter dust deposit was chosen as the best compromise between quartz assessment (thick dust deposit), gravimetric assessment and pump performance. Thicker and smaller diameter deposits would improve quartz sensitivity (in mg m^{-3} of air), but because of pump performance limitations they decrease airflow and degrade gravimetric sensitivity. Gravimetric sensitivity is limited by filter handling, slight damage or contamination in loading and unloading filter holders, rather than by balance sensitivity.

Bed and surface dust collection. One disadvantage of silver membrane filters is that the filter material is opaque to X-rays and any quartz dust collected in the body of the filter is not assessed by X-ray diffraction. At the start of sampling some 30% of the quartz was collected at depth and only 70% was 'seen' by the X-rays. As the dust bed builds up, the proportion collected in the body of the filter diminishes. This problem is discussed in the section on Calibration.

X-ray diffraction equipment

Cobalt Kα radiation is used, as it was found to be the best compromise between resolution and absorption.

A diffracted-beam bent-graphite-crystal monochromator with a scintillation detector and pulse-height analyzer were used, as they substantially improved count rate and signal to noise ratio compared with other techniques of eliminating interference by Kβ radiation.

An automatic sample changer is used with a standard in the first position and space for 34 samples. The filters are mounted in a turned well in the sample holders and held in place from the back with spring retaining rings.

Interference

A large number of minerals produce X-ray diffraction patterns in which one or more lines are close to those of quartz. Figure 3 shows interference with unresolved,

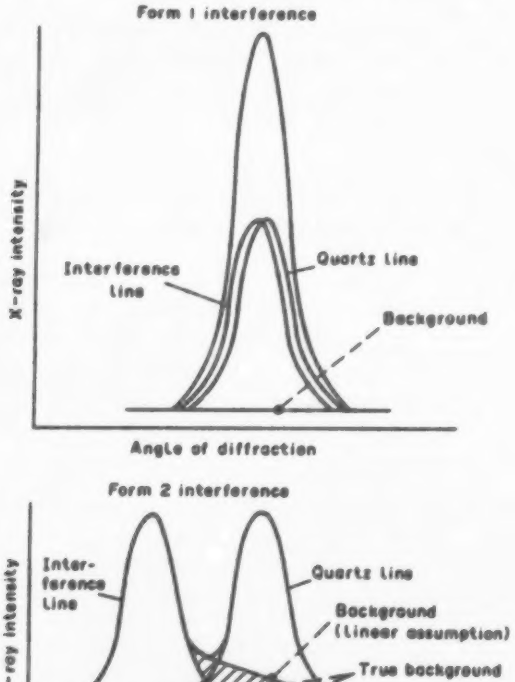
Form 1 interference
X-ray intensity
Interference line
Quartz line
Background
Angle of diffraction
Form 2 interference
X-ray intensity
Inter-ference line
Quartz line
Background (Linear assumption)
True background
Angle of diffraction

FIG. 3. Interference: effect of adjacent peaks.

form 1, and just resolved lines, form 2. In the first case the quartz is over-estimated. In the second it is under-estimated by the omission of the hatched area.

A simple check procedure has been chosen which is amenable to computer control. In this, the sample is assessed using two diffraction peaks. If the assessments agree within defined limits then the next sample is called for; if not, a third and maybe a fourth peak is analyzed. The flow diagram for computer control is shown in Fig. 4.

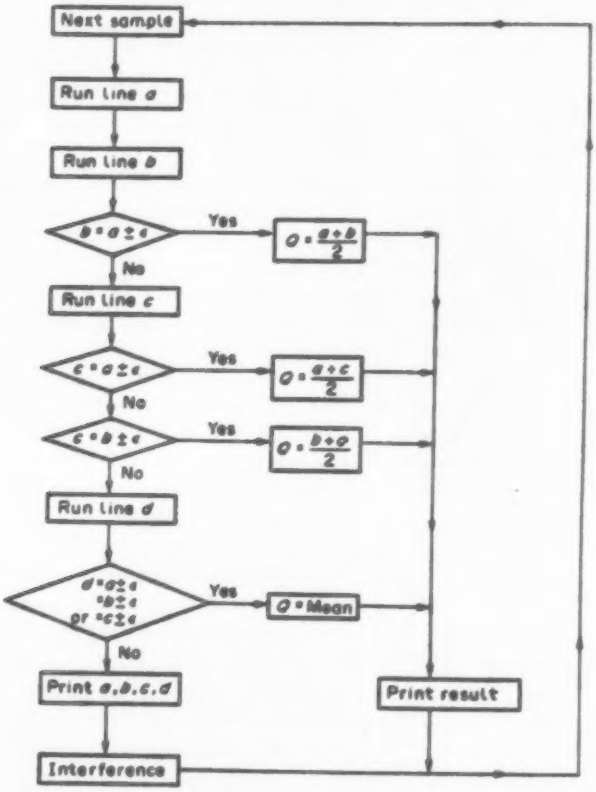
Next sample
Run line a
Run line b
b = a ± e
Yes
$Q = \frac{a+b}{2}$
No
Run line c
c = a ± e
Yes
$Q = \frac{a+c}{2}$
No
c = b ± e
Yes
$Q = \frac{b+c}{2}$
No
Run line d
d = a ± e
= b ± e
or = c ± e
Yes
Q = Mean
No
Print a, b, c, d
Print result
Interference

FIG. 4. Computer controls for interference.

Absorption

'Thin' samples are used to avoid the need to determine corrections for matrix and absorption effects (BRADLEY, 1967). The use of short wavelength X-rays decreases these problems, but short wavelength target tubes, such as molybdenum, produce lower count rates, lower signal to noise ratios, and less resolution than the longer wavelength tubes, such as copper. There are data available to calculate absorption for each mineral and for each X-ray target wavelength. If, arbitrarily, a maximum error of 20% due to uncorrected absorption is permitted, one can calculate dust weight limits. These are shown in Table 2 for one quartz line and four representative mine airborne dust

TABLE 2. CALCULATED MASS LIMITS WITHOUT ABSORPTION CORRECTION (quartz 1, 0, 1 line)

Constituent	Mass limit mg cm^{-2} X-ray target Mo	Cu	Co	Cr
SiO_2	6.8	2.1	1.4	0.82
$CaCO_3$	3.4	0.78	0.56	0.37
FeS_2	1.1	0.27	0.6	0.41
$H_{11}C_7O_8$ as O	30.0	10.0	7.0	5.0

constituents. This table suggests that cobalt radiation is a good choice. Copper radiation is not desirable because of the heavy absorption by iron, a common constituent of ores.

Absorption in the dust layer may be determined by measuring the intensity of the diffraction peaks of the silver substrate through it (LEROUX, 1972). This method works well on laboratory-prepared filters. There are, however, substantial variations in silver peak intensity between batches of filters, also field samples can apparently get 'tarnished', either from hydrogen sulphide gas or from reactions with the dust deposit. This tarnished layer of silver salts absorbs the X-rays very strongly and the method is not accurate. The errors due to absorption have exceeded 20% on less than 1% of the samples received. The method was tested in examining heavy samples, over 3 mg, in the Labrador iron ore study. This led to substantial corrections on some samples with weights above 5 mg. An approximate simple regression correction technique was written into the computer program.

Calibration

'Permanent' standards are used to correct for the secular variations in X-ray intensity that occur. The standard in daily use is checked at intervals against a set of standards. The standards consist of steel plates machined to fit the automatic sample chamber with a shallow well of the same diameter as the dust deposit on the filter. The wells are filled with a quartz resin mixture and ground flat. In calibration equivalent masses of quartz for the standards are determined by comparison of the areas under the peaks for four quartz diffraction lines with those obtained on weighed filters collected, using the appropriate sampler, in a pure quartz airborne dust cloud. The dust cloud is prepared by jet milling Ottawa (Illinois) silica sand (KNIGHT, 1978).

Figure 5 shows a schematic outline of a calibration graph. The XRD intensity/mass ratio is plotted against the mass of quartz on the filter. The top curve shows the calibration expected for pure quartz after allowing for absorption. The shape of this curve has been followed when using a number of filters, including 0.8 μm pore size silver membrane. The middle curve shows the calibration obtained with 1.2 μm pore size silver membrane filters. The lower curve shows that obtained with 5 μm pore size silver membrane filters, and the dashed line shows the calibration used for them. The dip on the left is believed to be due to the collection of quartz in the body of the filter, where it is hidden from the X-rays. As the dust bed builds up, this decreases and stops altogether at about a quartz weight of 0.4 mg on the 20 mm diameter dust deposit. The analytical procedure for samples on 5 μm filters is to determine the quartz, using the horizontal portion of the line, and to multiply the result by the light sample correction

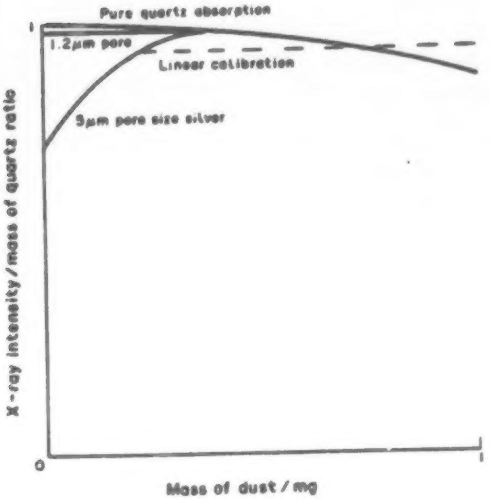

FIG. 5. Schematic outline of quartz calibration.

factor. This is based on the shape of the dip on the left and the weight of total respirable dust on the filter (KNIGHT, 1978). The weight of total respirable dust is used, because the quartz 'loss' is presumably related to the build-up of the total dust bed, not just the quartz portion.

The algorithm used is $Q_C = Q_M\ (1.4 - W)$ and $0 < W < 0.4$, where Q_C is quartz corrected, Q_M is quartz measured and W is the weight of the dust deposit in mg.

DISCUSSION

The Elliot Lake Laboratory has had the great advantage of having one individual responsible for both dust sampling and X-ray diffraction assessment for quartz. This has given that person the opportunity to develop compromises between sampling and assessment. Most quartz assessment laboratories have had to analyze samples with very limited opportunities for improving the sampling equipment.

The conflicting requirements of these different aspects have called for an integrated approach to the problem of developing, calibrating and testing a complete dust sampling and assessment system, and relating it to the environment, both technical and personnel, in which it is to be used. This is why the acronym CAMPEDS is now understood to mean Canadian Mines Personal Dust Sampling System, rather than the original sampler.

The CAMPEDS dust sampler has been used successfully by a number of mines. The success of the quartz assessment aspect can be judged by the throughput of our laboratory. In the course of one study some 8000 samples were submitted and one technician with one diffractometer achieved rates of 1200 samples per month.

This system, in common with all quartz assessment, has two major unsolved problems. Firstly, the form of quartz found in a mine may not match the reference quartz in crystallite size, distribution, orientation and X-ray intensity/mass ratio.

Laboratory experiments have found 50% differences in X-ray intensity/mass ratio from various quartz minerals, some of which may have contained up to 20% impurities (KNIGHT, 1978). Secondly, the human response to quartz may also differ considerably, depending on both the trace impurities in it and its admixture with other minerals (LE BOUFFANT *et al.*, 1982). Thus, the system should be backed up by regular medical lung X-rays with a reading system capable of measuring rates of progression, as has been used in the British coal mines (CRAWFORD et al., 1982). Dust target levels should be adjusted at intervals for each mine.

The statistics of sampling in the two underground mines examined show that dust concentrations (total, respirable quartz and combustible) are very variable from day to day, even for the same man doing the same job in the same working place (KNIGHT *et al.*, 1980). This study suggested that the difference between jobs could be significant, whereas between men and between areas for the same job the differences were not significant. More sampling would have been required, however, to determine if the differences between men or areas were significant and to give a feed-back into training or operating requirements.

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Section 1—Chapter 9

Comparison of Dust Samplers: Statistical Analysis Techniques

by

G. Knight and E. Moore

American Industrial Hygiene Association Journal
48(4) pp344-353 (1987)

COMPARAISON D'ÉCHANTILLONNEURS DE POUSSIÈRE : TECHNIQUES D'ANALYSE STATISTIQUE

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Une comparaison statistique de paires d'échantillonneurs de poussière a été menée à l'aide de cinq modèles : droite passant par l'origine, droite coupant les axes, modèle logarithmique, modèle logarithmique (poids + constante) et courbe forcée à passer par l'origine. Le quatrième et le cinquième modèles combinaient la partie arithmétique de l'erreur, associée à la pesée et à la manipulation, et la partie géométrique, associée à l'échantillonnage et aux variations du nuage de poussière, en une forme géométrique unifiée. On a montré que certaines des hypothèses sur lesquelles est basée l'analyse de régression par la méthode des moindres carrés n'étaient pas respectées et que des corrections étaient nécessaires. Dans la plupart des cas, on n'a pas noté de différences importantes entre les courbes de régression corrigées et la relation physique prévue, à savoir une droite passant par l'origine. Dans les exceptions, on a trouvé des raisons physiques expliquant l'absence de linéarité observée dans l'analyse statistique. Il appert que pour estimer la relation entre deux échantillonneurs de poussière, la meilleure méthode consiste à déterminer la droite passant par l'origine par la méthode des moindres carrés, en utilisant des variables transformées. Une méthode permettant de déterminer les valeurs des constantes est décrite. L'application de cette méthode statistique à des comparaisons d'échantillonneurs de poussière ne mesurant pas le même paramètre est examinée et un exemple est donné. Les considérations relatives à l'évaluation de mesures de poussière historiques sont soulignées.

Am. Ind. Hyg. Assoc. J. 48(4):344-353 (1987)

Comparison of Dust Samplers: Statistical Analysis Techniques

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Statistical analyses comparing pairs of dust samplers were carried out using five models, a straight line through the origin, linear with intercepts, logarithmic, a logarithmic (weight + constant), and a fifth forced through the origin. The fourth and fifth models combined the arithmetic portion of the error, associated with weighing and handling, with the geometric portion, associated with sampling and the dust cloud variations, into a unified geometric form. It was shown that some of the assumptions on which the least squares method of regression analysis are based were not met and that corrections had to be made. In most cases no significant difference was found between the corrected regression lines and the expected physical relation, a straight line through the origin. In the exceptions physical reasons were found to support the nonlinearity found in statistical analysis. It is suggested that the best estimate of the relation between two dust samplers can be obtained by a least squares determination of the straight line through the origin using transformed variables. A method is given for determining values of the constants. The application of this statistical method to comparisons between dust samplers not measuring the same parameter of the dust cloud is discussed with an example. The considerations in assessing historical dust measurements are outlined.

Nomenclature

Each mathematical model used can be obtained from this overall equation by omissions:

$$(y + C) = (Ax + X)^D + B$$

and for consistency and understanding all the equations have been written to include the constants in the form in which they appear here with subscripts corresponding to the equation number in the text.

A	is the slope of the expected relation between y and x.
A_1	is that of the line connecting the origin to the point $\bar{y}, \bar{x}$.
A_2	is the slope obtained in regression y upon x.
A_{14}	is the slope obtained in regression x upon y.
A_{22}	is the slope obtained in the regression of the transformed variable.
A_{23}	is the slope obtained in inverse regression.
A_{25}	is the slope of the line connecting the origin to the point ln(y+C), ln(x+C/A).
B	is the intercept on the y axis found in linear regression and is used in examining zero errors.
B_2	is that obtained in regression of y upon x.
B_{14}	is that obtained in regression x upon y.
C	is a constant used to combine the arithmetic and geometric portions of the errors in weight of samples.
D	is the power to which the variable x is raised in the regression equation used to examine nonlinearity.
D_{22}	is that obtained in regression on the transformation $Y = \ln(y+C)$.
D_{23}	is that obtained in inverse regression.
i	is the subscript identifying pairs of observations.
ln( )	is the natural logarithm of the expression enclosed in brackets.
N	is the number of pairs of observations included in analysis.
r	is the correlation coefficient.
r_2	is that obtained in linear regression.
r_{22}	is that obtained in linear regression on the transformed variable.
SE	is the standard error of the estimate of y found in analysis.
SE_2	is that in the linear regression.
SE_{22}	is that in the regression on transformed variables.
SE_{26}	is that of the straight line through the origin.
x	is the value (weight) of the reference dust sample.
X	is the transformed value used in regression analysis.
y	is the value (weight) given by the dust sampler being examined.
Y	is the transformed value used in regression analysis.
ϵ_i	is the error term for the ith pair being the difference between y and A_1x.
Σ	placed before a variable indicates summation over the N values of the variable.
$\bar{}$	is the bar symbol placed above a variable; *i.e.*, $\bar{y}$, indicates the arithmetic mean of the N values.

Introduction

Statistical techniques for comparing respirable dust samplers are developed in this paper to take into account the specific form and physical origin of the errors found. The techniques are applied to data collected in laboratory and field comparisons that are described in a separate paper.(1) Also given in the discussion are the physical bases of the comparisons between dust samplers not measuring the same parameter of

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the airborne dust and an example of the application of the statistical method developed.

It is expected that the relation between most pairs of dust samplers would be a straight line through the origin.

$$y = Ax \tag{1}$$

The objective of the statistics should be to make a best estimate of the slope of the relation and of the extent and statistical significance of any departure from the expected relation. Departure from the expected relationship can be separated into two cases: first, a zero error between the two instruments; and second, a non-linear or curved relationship. The linear regression equation

$$y = Ax + B \tag{2}$$

is useful in examining the first form of error with the intercept "B" being an expression of the zero error between the pair of samplers. A curved relationship could be represented and examined statistically in many ways. A common method is determining the significance of the coefficients in a power series as:

$$y = a_1x + a_2x^2 + a_3x^3 + a_nx^n + B. \tag{3}$$

An alternative is to use a logarithmic transformation:

$$Y = DX + \ln A \tag{4}$$

where, $Y = \ln y$, and $X = \ln x$

and apply linear regression to the transformed variables.

The development of an appropriate mathematical model should consider the physical cause of the nonlinearity.

Many authors have applied regression on a linear or logarithmic transformation with little if any discussion of the statistical requirements of the data base and the physical basis of the comparison, and they have presented the results without a discussion of the significance of the departures from a linear model.

The validity of regression analysis depends on meeting the assumptions on which the least squares method is based.(2)

Throughout this report dust samplers are referred to by name and cross-referenced by a code number to the detailed description given in the main paper.(1)

Studies of Variance

The assumptions used in least squares regression analysis(2) are as follows:

1) the average value of the errors is zero;
2) the errors have common variance;

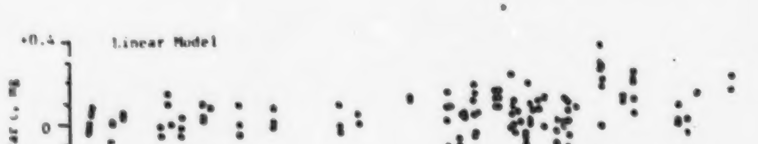

Figure 1 — Distribution of errors with linear, logarithmic and ln(y + C) models.

TABLE I
Comparison of Variance for the Various Models: CAMPEDS Sampler Laboratory Results

Samples	Mean Dust Weight y mg	No. of Pairs	Linear			Logarithmic (C=0)		Transformation Y = ln(y+C) (C=0.5)		(C=1.0)		(C=2.0)	
			r_{10}	SE mg	SE (CSE/$\bar{y}$)[A]	r_{16}	SE	r_{22}	SE	r_{22}	SE	r_{22}	SE
Comparison with Hexhlet													
Light	0.29	54	0.980	0.059	1.203	0.849	2.185	0.973	1.086	0.977	1.048	0.979	1.026
Medium	1.63	77	0.973	0.157	1.096	0.980	1.078	0.935	1.043	0.935	1.039	0.936	1.034
Heavy	5.80	20	0.936	0.274	1.047	0.935	1.046	0.935	1.043	0.935	1.039	0.936	1.034
Replication													
Light	0.29	50	0.977	0.063	1.217	0.889	2.092	0.965	1.099	0.972	1.054	0.968	1.029
Medium	1.63	71	0.960	0.184	1.113	0.975	1.087	0.982	1.057	0.948	1.077	0.985	1.030
Heavy	5.80	31	0.963	0.201	1.035	0.959	1.038	0.959	1.035	0.961	1.033	0.960	1.028

[A] This column has been calculated to illustrate the comparison between the often used coefficient of variation SE/Y, and the form of the standard error from Models 3 and 4.

3) the errors are independent;

4) the values of the independent variable are measured with negligible error;

5) the errors are assumed to follow a normal distribution.

For tests of significance the errors also are assumed to follow a normal distribution. These requirements are considered in order.

Systematic experimental errors would lead to Assumption 1) being broken, and it has to be appreciated that any systematic error in a particular study is not corrected for.

Assumption 2) that the errors have common variance can be checked by examining the errors separately in various ranges of the variable. This has been carried out by calculating the errors using the expected relation, Equation 1, for one pair of dust samplers.

$$\epsilon_i = y_i - A_1 x_i \qquad (5)$$

Figure 1 shows treatment of the error term for the pair of samplers with most replication in the laboratory study. The top of the figure shows the direct calculation for the linear case, and it can be seen that errors increase with x. The middle of the figure shows the effect of dividing ϵ_i by Ax_i, equivalent to a logarithmic transformation. It can be seen that this also does not give a satisfactory result. The bottom of the figure shows the effect of dividing ϵ_i by $(Ax_i + C)$. It can be seen that with this transformation the range of errors is almost constant over the range of x examined. This was studied further by determining the standard errors of the estimate of y from the regression equations from the expression:

$$S.E._a = [(1 - r^2)(\Sigma(y - \bar{y})^2)/(N - 2)]^{0.5} \qquad (6)$$

in the linear case and as the geometric ratio:

$$S.E. = e^{[(1 - r^2)(\Sigma(Y - \bar{Y})^2)/(N - 2)]^{0.5}} \qquad (7)$$

with the transformations:

$$Y = \ln y, \ X = \ln x \qquad (8)$$

$$Y = \ln(y + C), \ X = \ln(x + C/A) \qquad (9)$$

for the same sampler divided into three groups, light, medium and heavy, separately. A was calculated from Equation 9. Four values of the constant C were used in Equation 1 to examine the effect of changing it. This was determined for the replication as well as the relationship with the reference dust sampler. The results are shown in Table I. It can be seen that neither the linear nor the logarithmic models give uniform results in the three ranges, but that under the ln(y + C) transformation the error is reasonably constant, particularly when the highest value of C, 2.0, is used. These laboratory results were run under accurate control of dust conditions. In the field experiments the arithmetic portion of the errors associated with handling and weighing are usually close to the laboratory values, whereas the geometric portion is greater,* implying that a smaller value of the constant C should be used. An arithmetic distribution of errors was found when comparing replicate samplers with less than 0.2 mg of dust collected in mines or laboratory tests. This also could be determined by the use of "blanks." A logarithmic normal distribution of errors was found when comparing replicate samples with more than 1 mg of dust collected in mine or laboratory. The constant C is a physical representation of the unification of these two forms of error over the whole range of sample weights, and the ratio of the arithmetic standard error (from replicated light samples) to geometric standard error (from replicated heavy samples) is used. This is approximately 0.1 mg/0.1 = 1 mg for respirable dust by weight, and 0.01 mg/0.1 = 0.1 mg for respirable quartz dust.

It can be seen from Table I that the mean value of the geometric error also decreases with increasing values of C, and thus, for consistency in comparing the various samplers, either a specifically determined value or the same value should be used throughout. There is insufficient data available for most dust samplers to distinguish between their specific values and the values given above.

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*This presumably reflects the greater variability in dust clouds as presented to side-by-side dust samplers in mines than in the laboratory.

Regression analysis usually is applied to an equation in the form:

$$Y = AX + B \qquad (10)$$

where, Y and X are functions of transformations of the measured variables y and x. This usually is understood to give the best estimate of Y for a known X. Assumption 4, however, is not met insofar as significant errors occur in the measurements of the "reference" sampler, X.

Davies has discussed the treatment when the independent variable is measured with significant error.(3) He suggests that the inverse regression

$$X = (1/A')Y + (-B'/A') \qquad (11)$$

also should be run and that an intermediate line based both upon the expected physical relationship and weighting by the respective variances of X and Y be obtained and assume a "functional" relationship between Y and X. He suggests that the slope A of the functional relation be obtained as follows:

$$A = m + (m^2 + k^2)^{0.5} \qquad (12)$$

where, $$m = \frac{1}{2A_{10}} - \frac{k^2}{2A_{11}}$$

and, $$k^2 = \sigma^2_Y/\sigma^2_X \qquad (13)$$

σ^2_Y and σ^2_X are, respectively, the variances of Y and X.

There is insufficient replication in most cases to determine the variances of the instrument and reference measurements. Thus, the "functional" relation cannot be determined precisely. The best estimates for the "slopes" and "intercepts" must lie between the values calculated by direct and inverse regression, Equations 10 and 11.(3)

Mathematical Models and Treatment

The expected relation between most pairs of dust samplers is Model 1:

$$y = A_1x \qquad (1)$$

The objective of the statistical treatment is to make a best estimate of the slope, A_1, and to determine the significance and extent of departures from the relation using least squares methods.

The second model considered is useful for determining the zero error, or intercept, between the pairs of samplers. The form often used in experiments is

$$y = A_2x + B_2. \qquad (2)$$

Following Davies, the inverse form also is subjected to regression analysis

$$x = \frac{1}{A_{11}}y - \frac{B_{14}}{A_{14}}. \qquad (14)$$

These models do not meet the requirement for common variance. Following Draper and Smith,(1) common variance can be achieved by dividing each value by $(C + x_i)$.

$$Y_i = y_i/(C+x_i);\ X_{1,i} = x_i/(C+x_i);\ X_{0,i} = 1/(C+x_i) \qquad (15)$$

[Equation 15 could be expanded to handle curvature by adding $X_{2,i} = X^2/(C+x_i)$] to give Model 3

$$Y = A_{16}X_1 + B_{16}X_0 \qquad (16)$$

and its inverse

$$X_1 = A_{17}Y_1 + B_{17}X_0. \qquad (17)$$

All the analyses carried out using Model 2 (Equations 10 and 11), however, showed that the intercept was insignificant, and no analyses were carried out using Model 3.

Many models could be considered for examining for curvature. The form chosen is a power law

$$y = Ax^D. \qquad (18)$$

This is put into a suitable form by logarithmic transformation

$$Y = \ln y, \text{ and } X = \ln x$$

to give Model 4

$$Y = D_{19}X + \ln A_{19} \qquad (19)$$

gives $y = A_{11} X^{D_{19}}(1+e)$; so that the error is $A_{19} D_{19} e$ where e is random and independent of X. The inverse is:

$$X = D_{20}Y + (-\ln A_{20}). \qquad (20)$$

If the variance of the model of Equation 18 is taken to be non-constant, then linear regression yet can be carried out by dividing each value by $(C + x_i)$ as in Model 3:

$$y_i/(C+x_i) = A(x_i)^D/(C+x_i). \qquad (21)$$

For the present paper, however, an alternative approximate solution was obtained using the transformation.

$$Y = \ln(y+C) \text{ and}^* X = \ln(x+C/A')$$

$$\ln(y+C) = D_{22}\ln(x+C/A') + \ln A_{22} \qquad (22)$$

$$\ln(x+C/A') = 1/D_{23} \ln(y+C) + (-\ln A_{23}) \qquad (23)$$

These two equations were solved by inserting the value of A_1 from Equation 1 for A' in Equations 22 and 23. The analysis then could be repeated using A_{22} and A_{23} for A'. The differences were small, however, and no reiterations were carried out.

Equations 22 and 23 were used first to determine the values of the power D_{22} and D_{23} to which x is raised. It was found that the values of D_{22} and D_{23} were usually close to unity. The significance of the power, D, can be determined using Equation 7.

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*It can be shown that C should be divided by A in this transformation by plotting the exact relations such as y - 2x, y+C = 2(x+C)

TABLE II
Results of Analyses: Gravimetric Assessments in Laboratory Tests

Code No.	Dust Sampler	No. of Pairs	Slopes						Intercept with y axes		Power		Correlation Coefficient		Standard Error of Estimate		
			Model 1	Model 2		Model 5		Model 6	Model 2, mg		Model 5		Model 2	Model 5	Model 2	Model 5	Model 6
		N	A_1	A_{10}	A_{11}	A_{22}	A_{23}	A_{25}	B_{10}	B_{11}	D_{22}	D_{23}	r_{11}	r_{22}	SE_2	SE_5	SE_6
Elliot Lake Reference Sampler Hexhlet																	
4	NCB MRE	56	0.0369	0.0375	0.0376	0.0390	0.0388	0.0363	-0.03	-0.04	0.976	0.980	0.998	0.998	0.11	1.039	1.041
	CAMPEDS																
5	Heavy samples only	28	0.0354	0.0342	0.0390	0.0427	0.0321	-	0.20[A]	0.59[A]	0.928	1.061	0.936	0.935	0.27	1.039	-
	Light and medium samples	130	0.0387	0.0380	0.0384	0.0392	0.0390	0.0387	0.02	0.01	0.983	0.993	0.995	0.995	0.09	1.043	1.044
	All	158	0.0369	0.0352	0.0354	0.0401	0.0396	-	0.09	-0.08	0.957[B]	0.962[B]	0.997	0.998	0.15	1.044	-
6	ACGIH flow rate	27	0.0404	0.0382	0.0399	0.0428	0.0416	0.0407	0.05	0.01	0.943	0.962	0.998	0.991	0.17	1.062	1.067
7	BCIRA #3	57	0.0385	0.0388	0.0391	0.0387	0.1384	0.0385	-0.02	-0.03	0.996	0.999	0.996	0.998	0.19	1.040	1.041
7	BCIRA #5	57	0.0421	0.0425	0.0427	0.0415	0.0415	0.0419	-0.02	-0.03	1.008	1.011	0.998	0.999	0.15	1.032	1.032
8	ACGIH flow rate	23	0.0418	0.0415	0.0431	0.0378	0.0377	0.0417	-0.02	-0.01	1.007	1.011	0.998	0.998	0.21	1.049	1.050
	Nylon cyclone																
9	25-mm metal FH	54	0.0243	0.0236	0.0238	0.0148	0.0246	0.0244	0.04	0.01	0.983	0.991	0.994	0.996	0.15	1.048	1.048
10	Plastic with sleeve	111	0.0210	0.0213	0.0218	0.0211	0.0206	0.0210	-0.02	-0.04	0.992	1.030	0.988	0.981	0.19	1.101	1.101
11	Plastic with nipple	26	0.0193	0.0189	0.0200	0.0196	0.0192	0.0193	0.09	-0.02	0.966	1.000	0.972	0.982	0.10	1.050	1.054
12	37-mm plastic with sleeve	24	0.0206	0.0204	0.0213	0.0204	0.0197	0.0205	0.01	-0.08	1.003	1.035	0.978	0.984	0.25	1.080	1.080
13	37-mm plastic with sleeve	49	0.0195	0.0199	0.0204	0.0194	0.0190	0.0194	0.02	-0.04	1.004	1.036	0.987	0.984	0.16	1.083	1.083
	CMDS																
14	Type I																
16	Modified for quartz	27	0.0200	0.0192	0.0210	0.0205	0.0198	0.0200	0.02	-0.02	0.956	1.033	0.957	0.962	0.12	1.086	1.087
17	Type II																
18	Modified for quartz	26	0.0201	0.0190	0.0240	0.0219	0.0196	0.0201	0.02	-0.08	0.901	1.086	0.890	0.911	0.21	1.120	1.127
Other Laboratories																	
	Limestone reference mean of #3, 14 and 16																
3	NCB MRE	23	1.324	1.334	1.347	1.337	1.212	1.322	0.04	0.09	0.993	0.999	0.995	0.997	0.35	1.065[C]	1.066
14	CMDS	45	0.877	0.896	0.926	0.873	0.852	0.873	-0.07	-0.18	1.001	1.019	0.984	0.991	0.35	1.076	1.076
16	CMDS 1.2 L/min	46	0.795	0.764	0.796	0.826	0.803	0.798	0.12	0.00	0.974	0.996	0.980	0.989	0.33	1.079	1.080
	Coal dust reference mean of #3, 14 and 16																
3	NCB MRE	18	1.415	1.461	1.478	1.361	1.338	1.403	-0.11	-0.14	1.023	1.038	0.994	0.993	0.24	1.070[C]	1.071
14	CMDS	20	0.821	0.790	0.805	0.855	0.844	0.824	0.07	0.03	0.962	0.976	0.990	0.993	0.14	1.047	1.049
16	CMDS 1.2 L/min	60	0.755	0.712	0.737	0.787	0.758	0.757	0.10	0.04	0.961	0.999	0.983	0.981	0.14	1.059	1.059
	Limestone and coal replicates																
16	CMDS	24														1.068	
16	CMDS 1.2 L/min	99														1.058	

[A] IS intercept significant

[B] SS slope significantly different from unity

[C] The values given here assume that there is agreement between the horizontal elutriators used in the two laboratories

In most cases the differences between the calculated slopes and a straight line through the origin (D=1) were not significant. Then the most likely value for the slope of the expected relationship between the pair of dust samplers, A in Equation 1, can be obtained from Model 6:

$$\ln(y+C) = \ln(x+C/A') + \ln(A_{25}) \quad (24)$$

as

$$A_{25} = e^{[\Sigma\ln(y+C) - \Sigma\ln(x+C/A')]/N} \quad (25)$$

where A' may be estimated by A_1 from Equation 1.

The standard error of y in the geometric form can be obtained as follows:

$$SE_{16} = e^{\text{[illegible]}} \quad (26)$$

to give Model 5 with its two equations.

Results of Statistical Analysis

Tables II to V show the results of the four models in comparisons between dust samplers carried out in the laboratories and in mines: simple relation through the origin (Equation 1); linear regression (Equations 10 and 11); ln(y+C) transformation (Equations 22 and 23); and the ln(y+C) forced through the origin (Equation 24). The organization of these studies was that two or more respirable dust samplers were operated side by side for the same period. Each respirable dust sampler was assessed by weighing for total respirable dust, and some samples were assessed by X-ray diffraction for respirable quartz dust. The "reference" sampler, the independent variable x, was chosen as a high volume sampler in some of the laboratory tests or as a mean given by measurements of some or all of the samplers in each study. The reference sampler is listed in Tables II to V.

It can be seen in Tables II to V that the six values for the slope A can be fairly close or can be widespread. The intercepts B are usually small and insignificant. The powers D are usually close to unity. Because the independent variable X, the reference dust sampler, is not measured with negligible error, a line intermediate between the regressions of Y upon X and X upon Y should be chosen as the best representation of the relation between the two dust samplers.(3) The correlation coefficients are all highly significant, and the standard errors differ substantially.

Figures 2 and 3 show, respectively, on linear and logarithmic scales the six relationships derived from Equations 1, 10, 11, 22, 23 and 26 for the two instruments showing the least and the greatest spread of variability about the reference sampler in the main laboratory experiment.

It can be seen that for the sampler with the least variability, the CAMPEDS, the maximum differences between the six lines is very small — less than the greatest of 0.02 mg or 1%. For that with the most variability, the coal mine dust sampler, the spread is greater with the lines through the origin lying between the pairs of lines derived from regression in both directions. It is concluded that the line through the origin, Equation 1, is an expected and reasonable assumption for the relation between the two samplers and that the best estimate of its slope is derived satisfactorily from Equation 26. The estimate possibly could be improved be rejecting outliers found when applying Equation 23 to the data. The data rejected in these studies have been selected on the basis of both reported faults in sampling or handling, and outlying by more than three standard deviations.

The results for the relation between quartz as measured on the X-ray diffractometer on each sampler with weight on the reference sampler show a fairly consistent positive value for the power. This probably is due to a systematic error in XRD assessment because of a small uncorrected absorption effect in the samples.

Discussion

It is shown that the assumptions on which statistical regression is based are not met in this data on comparisons between dust samplers measuring almost the same parameter of dust clouds. The suggested solution is the determination of a transformation chosen to give uniform errors over the range of the measurements, followed by regression analyses of X upon Y as well as the desired Y upon X. It is shown that the differences between the regression lines obtained by the Models 1 and 6 are intermediate to the values obtained by

TABLE III

Results of Analyses: Quartz Assessments in Laboratory Tests

			Slopes						Intercept with y axes		Power		Correlation Coefficient		Standard Error of Estimate		
Code No.	Dust Sampler	No. of Pairs N	Model 1 A_1	Model 2 A_{10}	A_{11}	Model 5 A_{22}	A_{23}	Model 6 A_{25}	Model 2 (mg) B_{10}	B_{11}	Model 5 D_{22}	D_{23}	Model 2 r_{10}	Model 5 r_{22}	Model 2 SE.	Model 5 SE	Model 6 SE..
	Horizontal elutriator																
3	113A	20	0.0300	0.0349	0.0355	0.0265	0.0262	0.0271	-0.067	-0.075	1.052	1.071	0.992	0.991	0.088	1.147	1.157
	CAMPEDS																
5	Light and medium	52	0.0387	0.0404	0.0406	0.0372	0.0369	0.0375	-0.019	-0.022	1.026	1.027	0.997	0.997	0.049	1.081	1.084
6	ACGIH flow rate	20	0.0450	0.0462	0.0472	0.0421	0.0345	0.0428	-0.018	-0.031	1.042	1.049	0.990	0.996	0.134	1.101	1.128
9	BCIRA #3	18	0.0383	0.0416	0.0420	0.0361	0.0354	0.0370	-0.031	-0.034	1.034	1.039	0.996	0.997	0.051	1.081	1.095
	BCIRA #5	20	0.0413	0.0442	0.0445	0.0370	0.0365	0.0382	-0.047	-0.043	1.055	1.063	0.998	0.996	0.056	1.099	1.118
	Nylon cyclone																
9	25-mm metal FH	20	0.0202	0.0240	0.0246	0.0184	0.0181	0.0186	-0.053	-0.062	1.032	1.066	0.987	0.984	0.078	1.176	1.179
10	Plastic with sleeve	40	0.0218	0.0254	0.0264	0.0199	0.0192	0.0196	-0.050	-0.063	1.041	1.088	0.982	0.987	0.094	1.210	1.214
11	Plastic with nipple	18	0.0265	0.0301	0.0304	0.0243	0.0238	0.0244	-0.045	-0.048	1.044	1.063	0.995	0.991	0.061	1.141	1.479
12																	
13	37-mm plastic with nipple	19	0.0117	0.0134	0.0139	0.0111	0.0111	0.0112	-0.024	-0.030	1.027	1.047	0.983	0.990	0.052	1.108	1.110
	CMDS																
15	Type I Mod. for Quartz	20	0.0098	0.0111	0.0113	0.0094	0.0094	0.0095	-0.017	-0.020	1.030	1.068	0.992	0.982	0.029	1.138	1.140
17	Type II Mod. for Quartz	19	0.0075	0.0091	0.0098	0.0066	0.0071	0.0072	-0.022	-0.032	1.015	1.074	0.963	0.972	0.051	1.154	1.154

ABLE IV
Results of Analyses: Gravimetric Assessments in Field Tests

Code No.	Dust Sampler	No. of Pairs N	Slopes Model 1 A_1	Slopes Model 2 A_{10}	Slopes Model 2 A_{11}	Slopes Model 5 A_{22}	Slopes Model 5 A_{21}	Model 6 A_6	Intercept with y axes Model 2 (mg) B_{10}	Intercept with y axes Model 2 (mg) B_{11}	Power Model 5 D_{22}	Power Model 5 D_{21}	Correlation Coefficient Model 2 r_{10}	Correlation Coefficient Model 5 r_{22}	Standard Error of Estimate Model 2 SE	Standard Error of Estimate Model 5 SE	Standard Error of Estimate Model 6 SE
	Test 1 reference mean of # 5 and 7																
3	NCB MRE	67	1.576	1.234	1.644	1.488	1.420	1.604	0.10	-0.02	1.220[a]	1.414[a]	0.867	0.929	0.15	1.173	1.192
5	CAMPEDS	75	1.070	1.048	1.074	1.188	1.068	1.070	0.01	0.00	0.976	1.006	0.988	0.985	0.10	1.028	1.028
7	BCIRA	76	0.926	0.916	0.944	0.924	0.918	0.924	0.00	-0.01	1.003	1.035	0.986	0.985	0.03	1.026	1.026
	Test 1 analysis of included replicates																
3	NCB MRE	23	-	-	-	-	-	-	-	-	-	-	-	-	0.18	1.101	
5	CAMPEDS	30	-	-	-	-	-	-	-	-	-	-	-	-	0.06	1.044	
7	BCIRA	31	-	-	-	-	-	-	-	-	-	-	-	-	0.05	1.034	
	Test 2 reference means # 4, 5 and 7																
4	NCB MRE	31	0.903	0.917	0.923	0.931	0.938	0.901	-0.01	-0.01	1.016	1.021	0.997	0.998	0.05	1.024	1.025
5	CAMPEDS	61	1.027	1.010	1.020	0.997	1.011	1.028	0.00	0.00	0.983	0.992	0.995	0.996	0.06	1.032	1.032
7	BCIRA	33	1.017	1.020	1.022	1.015	1.019	1.017	0.00	0.00	0.999	1.001	0.999	0.999	0.03	1.016	1.016
10	Nylon 25 mm	16	0.601	0.529	0.549	0.520	0.509	0.605	0.05	0.14	0.891	0.920	0.981	0.984	0.07	1.045	1.055
12	Nylon 37 mm	17	0.621	0.568	0.582	0.557	0.575	0.624	0.03	-0.03	0.931	0.962	0.988	0.984	0.06	1.048	1.052
	Test 3 reference # 7A BCIRA																
12A	Nylon 37 mm	29	0.600	0.593	1.071	0.585	0.624	0.602	0.00	-0.17	0.980	1.615	0.797	0.779	0.36	1.236	1.251
	Test 4 reference mean of # 4, 5A, 7A and 12A																
4	NCB MRE	64	0.968	0.887	0.967	0.846	0.908	0.981	0.07	-0.01	0.945	0.982	0.958	0.982	0.17	1.059	1.062
5A	CAMPEDS	64	1.135	1.087	1.105	1.072	1.116	1.137	0.03	-0.02	0.979	0.996	0.992	0.992	0.09	1.042	1.043
7A	BCIRA	68	1.224	1.451	1.508	1.400	1.764	1.204	-0.16	-0.20	1.077	1.104	0.981	0.988	0.18	1.059	1.065
12A	Nylon 3 mm	67	0.680	0.579	0.700	0.536	0.900	0.682	0.07	-0.02	0.902	1.070	0.903	0.918	0.17	1.101	1.104
	Test 5 reference instrument # 7A (BCIRA)																
5A	CAMPEDS	414				1.968	1.641	-			0.885[a]	0.913[a]		0.984		1.068	-
	Test 6 reference mean of # 3, 14 and 15																
3	NCB MRE	57	1.529	1.542	1.651	1.524	1.304	1.527	-0.008	0.070	1.001	1.058	0.966	0.973	0.127	1.059	1.059
14	CMDS	55	0.724	0.693	0.752	0.787	0.419	0.716	0.014	-0.021	0.967	1.046	0.960	0.961	0.067	1.045	1.045
15	CMDS (1.2 L/min)	57	0.748	0.771	0.836	0.711	0.591	0.725	-0.013	0.050	1.020	1.097	0.960	0.964	0.071	1.045	1.045

[a] SS slope significantly different from unity.

direct and inverse regression with Models 2 and 4. There is insufficient replication to calculate variances of X and Y to follow Davies' approach rigorously in most cases.(3) This approach, however, suggests that the difference between a statistical estimation of the "functional" relation and the straight line through the origin, Model 6, is not significant for the few cases examined. It is suggested that Model 6 is the best estimate of the relation both for the existing data and for extrapolation, unless a physical reason can be found for the apparent nonlinearities indicated by the analysis.

The dangers of extrapolation are demonstrated in the results from the CAMPEDS dust sampler in which linearity was found in its original testing, and for light and medium samples. Here, nonlinearity was suggested in a comparison with the B.C.I.R.A. cyclone in a field comparison and confirmed in laboratory testing in an extended range (heavy samples). In the extended range testing, a physical cause for the nonlinearity was identified.

While the authors have discounted intercepts in this study, it is possible that they could exist. The normal linear regression technique, however, can produce them as an artifact, as shown in Figures 2 and 3. Presumably this is an effect of the errors in the independent variable. As a first step the weighted mean of the intercepts obtained in the regressions of Y on X and X on Y, Equations 16 and 17 should be determined.(3) Then the statistical and physical significance should be considered. Figure 3, for instance, suggests that an intercept of about 0.03 mg could exist in the upper comparison. This might be attributed to systematic losses of fiber from the glass fiber filters used in the reference sampler. The lower curve in Figure 3 shows that intercepts ranging from -0.05 to +0.05 can be found for the various models; however, it can be seen that the mean value from the regressions is near to zero.

The comparison of dust samplers not measuring the same parameter of the dust cloud is more difficult, especially when the major objective is to extrapolate to higher dust levels for interpreting past dust exposures in terms of measurements that would be given by presently used instruments. Attempts at this have used sets of side-by-side measurements by the old and new instruments in the existing workplaces. Because of the often very great improvements in dust control achieved in the years when the old samplers were in use, substantial extrapolation often is needed and single sided regression techniques could give rise to large errors. Clearly, it is desirable to recreate the old operating techniques and high dust levels and carry out direct comparisons in the range, but in view of the difficulties this has been reported only once.(8) Thus, it is necessary to attempt to understand the physical relation between the pair of samplers. A start in this is understanding of the parameters of the dust cloud and the probable changes in them as dust control has improved and mining (operating) techniques and equipment have changed. On the basis of physiology and aerodynamic behavior, airborne particles are considered in five size groups:

Very coarse	– non-inhalable;
Coarse	– nasal deposition;
Medium	– tracheo-bronchial deposition;
Fine	– alveolar deposition "respirable dust";
Very fine	– smokes and fumes whose behavior and deposition are controlled by diffusion.

Airborne particulates in mines have four main origins:

1) Mineral breakage, leading to substantial quantities in the coarse to fine sizes and abbreviated as VC, C, M and F, respectively.
2) Internal combustion engine exhaust, giving substantial quantities mostly in the very fine size range and referred to as IC.
3) Compressed air powered equipment, giving substantial quantities of lubricating oil mist in medium and fine size ranges, referred to as OM. This oil mist is collected on filters and contributes to the weight in most gravimetric sampling.
4) Air pollution, entering with mine intake air in fine and very fine size ranges (AP).

In addition, some specific components of the mineral dust need to be given special consideration because of their high potential health hazard. The most common two are quartz (Q) and mineral fiber (MF), including asbestos, in the fine size range.

Historically, as dust control has improved and mining methods have changed, the major changes in dust cloud parameters can be characterized as follows: as a reduction in mineral dust concentrations in all size ranges, with more reduction for the coarser than the finer dusts; as the substantially increasing proportion of IC engine exhaust as their use has increased and the mineral dust reduced; as changing proportion of oil mist with equipment changes and mineral dust concentrations; and as, in some cases, proportionally significant quantities of air pollution when mineral dust has to be reduced to very low levels as in preparation of silica flour.

Dust sampler response is usually a single parameter, weight or count with some size selection and can be considered as the sum of the responses to the individual components of the dust cloud:

$$R = VC + C + M + F + Q + MF + IC + OM + AP \qquad (27)$$

where R is the total sampler response;

VC is the response to the very coarse mineral, *etc.*

The worker also can be considered as a dust sampler with the response measured by health effects using this equation and enabling a weighting to be applied to each component.

Often one component is of major significance, and an instrument that gives specific response to that component is desired – for instance, assessment of respirable sized quartz using X-ray diffraction in gold mines or fiber count in asbestos mines and mills.

The non-specific instrument often responds to the mineral and non-mineral components of the dust cloud. The non-mineral, such as air pollution, often increases in proportion as the dust cloud concentration drops, leading to increased error in the comparison at low concentrations, and if the experimental data covers the range of interest, the statistical approach given here may well produce the best estimate of

TABLE V
Results of Analyses: Quartz Assessments in Field Tests

Code No.	Dust Sampler	No. of Pairs N	Slopes Model 1 A_1	Slopes Model 2 A_{10}	Slopes Model 2 A_{11}	Slopes Model 5 A_{20}	Slopes Model 5 A_{21}	Slopes Model 6 A_{2P}	Intercept with y axes Model 2 (mg) B_{10}	Intercept with y axes Model 2 (mg) B_{11}	Power Model 6 D_{20}	Power Model 6 D_{21}	Correlation Coefficient Model 2 r_{10}	Correlation Coefficient Model 5 r_{20}	Standard Error of Estimate Model 2 SE_2	Standard Error of Estimate Model 5 SE	Standard Error of Estimate Model 6 SE_6
	Test 1: reference mean of # 5 and 7																
5	CAMPEDS	70	1.104	1.106	1.148	1.094	1.160		0.002	0.000	0.972	1.004	0.981	0.984	0.010	1.054	1.061
7	BCIRA	72	0.882	0.907	0.969	0.930	1.080		-0.001	-0.004	1.027	1.074	0.968	0.978	0.012	1.060	1.065
	Test 1 analysis of included replicates																
5	CAMPEDS	30	-	-	-	-	-		-	-	-	-	-	-	0.011	1.071	
7	BCIRA	28	-	-	-	-	-		-	-	-	-	-	-	0.017	1.084	
	Test 2: reference mean # 4, 5 and 7																
4	NCB MRE	31	0.830	0.831	0.834	0.836	0.869		0.000	-0.002	1.014	1.016	0.998	0.999	0.032	1.055	1.060
5	CAMPEDS	61	1.058	1.045	1.052	1.024	1.035		0.005	-0.002	0.986[A]	0.989[A]	0.997	0.998	0.051	1.059	1.066
7	BCIRA	35	1.050	1.040	1.045	1.042	1.053		0.000	-0.003	1.002	1.005	0.998	0.999	0.048	1.063	1.068
10	Nylon 25 mm	17	0.444	0.453	0.470	0.403	0.440		-0.004	-0.013	0.973	0.997	0.981	0.988	0.061	1.143	1.155
12	Nylon 37 mm	17	0.279	0.287	0.289	0.277	0.293		-0.004	-0.005	1.004	1.015	0.996	0.995	0.018	1.077	1.086
	Test 4: reference mean # 4, 5A, 7A and 12A																
4	NCB MRE	64	0.922	1.004	1.043	1.029	1.103	0.911	-0.005	-0.007	1.066	1.108	0.981	0.981	0.014	1.071	1.074
5A	CAMPEDS	62	1.182	1.115	1.138	1.134	1.189	1.186	0.003	-0.002	0.997	1.001	0.990	0.988	0.010	1.056	1.057
7A	BCIRA	63	1.141	1.136	1.163	1.224	1.167	1.137	0.000	-0.001	1.014	1.040	0.988	0.987	0.012	1.061	1.062
12A	Nylon 37 mm	66	0.292	0.260	0.361	0.221	0.374	0.292	0.002	-0.004	0.841	1.202	0.848	0.836	0.011	1.083	1.087
	Test 5: reference instrument # 7A (BCIRA)																
5A	CAMPEDS	416	-	-	-	1.126	0.879	-	-	-	0.980	1.029	-	0.976		1.098	-

[A] SS slope significantly different from unity.

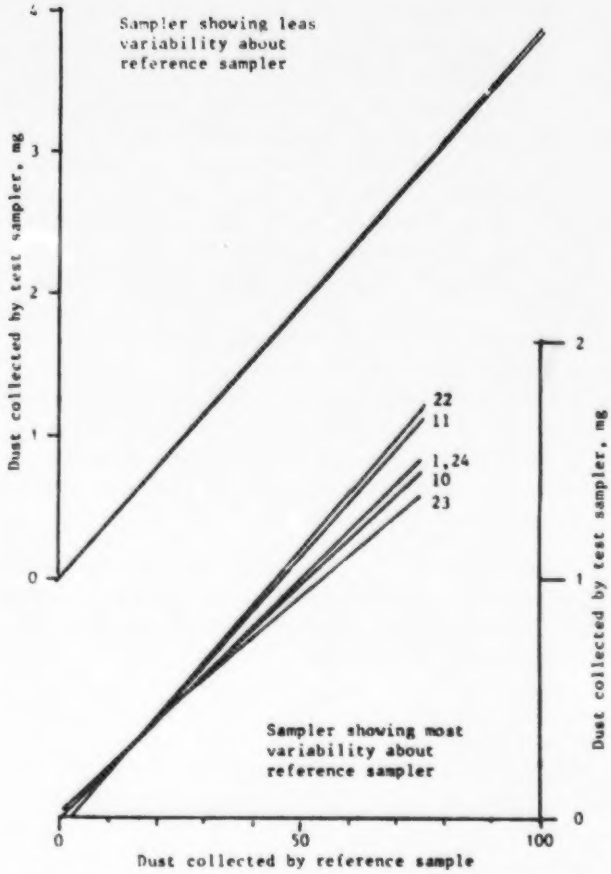

Figure 2 — Lines given by the regression analyses: linear scale.

the relation between the two samplers and the estimate of error in this relation.

When it also is required to extrapolate to higher past dust levels, the use of a function other than the straight line through the origin is likely to lead to large errors unless it is supported by experiment and/or physical reasoning.

The linear assumption requires that the experimental data extend into dust concentrations high enough for mineral to be the major component and that the ratio of the responses of the two samplers is not affected by the historical changes, if any, in size distribution and composition of the airborne mineral dust.

These techniques, Equations 22-26, were applied to 152 pairs of side-by-side dust measurements obtained with 90-min membrane filter samples, fiber count, and the mean of 10 to 20 evenly spaced 4-min respirable dust samples with the weight determined by beta absorption. The data are shown in Figure 4. These points were divided into approximately equal parts by a line drawn at right angles to the trend, as shown. Models 5 and 6 were applied with various values of the constant C to the low and high sets of data to give the following estimates of the standard error and the correlation coefficient.

Constant C		Standard Error of Estimate		Correlation Coefficient	
Fibers/mL	mg/m^3	Low Set	High Set	Low Set	High Set
0.0	0.0	1.73	1.60	0.04	0.76
0.2	0.037	1.60	1.56	0.03	0.76
0.3	0.056	1.55	1.55	0.03	0.77
0.5	0.093	1.47	1.52	0.02	0.77
1.0	0.185	1.36	1.48	0.03	0.77

It can be seen that the low data set gives no correlation between the two measurements, whereas for the high set this is highly significant. The best agreement between the standard errors of estimate for the two sets is with the constant C set at 0.3 fibers/mL and 0.056 mg/m^3. When this is used, the results of regression of Y on X and X on Y are shown in Figure 3 (Equations 22 and 23). It can be seen that the line through the means and the origin almost bisects these lines. Clearly, the possible errors in extrapolation are large and the most likely estimate for extrapolation is Model 6, a straight line through the origin and the point X, Y, with a geometric standard estimate of error of 1.71 in the experimental range. This "error" is believed to be partly due to real fluctuations in fiber and size distributions in both space and time. Model 1 is not desirable, as it gives a higher weighting to the results from the low range where there is a lot of interference from non-mineral dust.

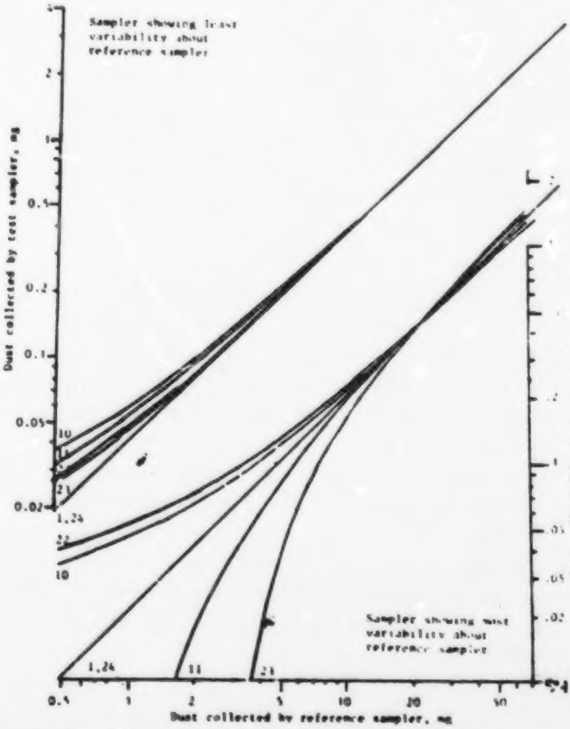

Figure 3 — Lines given by the regression analyses: logarithmic scales.

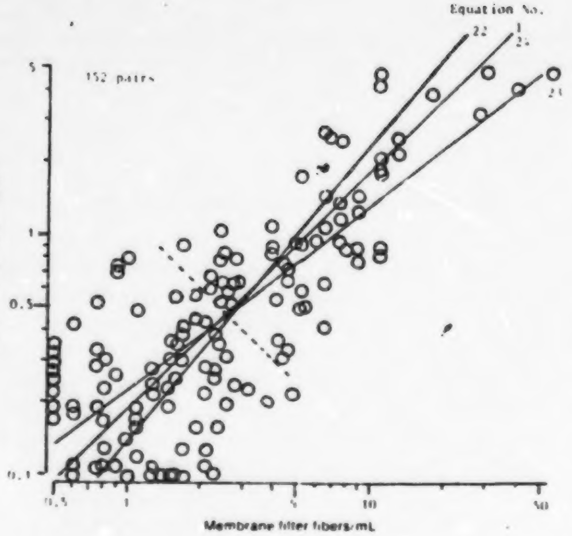

Figure 4 — Comparison of two samplers assessing dust by different parameters.

Presumably, at low fiber dust concentrations, the respirable dust sampler is measuring appreciable proportions of non-mineral dust from unrelated dust sources.

It was noted that Du Toit *et al.* formally used the transformation Y=ln(y+C); however, this was not justified on physical grounds, but on mathematical grounds to remove the possibility of introducing infinities.(6) The authors have avoided these by setting zero and negative values to a value at or below the detection limit of 0.01 mg for weight and 1 μg for quartz. They used the same value, C=1, in defining both X and Y; thus, if the mean ratio of x to y was not unity, they introduced nonlinearity into the expected relation and apparently did not deal explicitly with the problem of significant error in the independent variable.

Conclusions

1. The assumptions on which the least squares method of linear regression is based are not met, and corrective action has to be taken.(2-4)
2. The relation between two size selective gravimetric dust samplers usually can be represented by a straight line through the origin.
3. The slope of this line can be determined best from the ratio of the means Y/X of the transformed dust weights, Equation 24.
4. The best estimate of error is given by considering the weight of dust and estimating the geometric standard error derived from Equation 18 using the transformation Y=ln(y+C).
5. The constant C, in general, can be obtained by splitting the data into groups, and by trial and error, finding a value that will give the most consistent error term over the groups. In this study, however, a uniform value was used in all the comparisons, estimated from the results of replicate measurements in mines. The values used were 1.0 mg for the weight of respirable dust on the filter and 0.1 mg for the weight of respirable quartz on the filter.
6. To avoid transforming a straight line into a curved relation, a derived constant C/A in the transformation of the reference sampler has to be used, ln(x+C/A).
7. A few nonlinear relations between pairs of dust samplers were found; these and their physical explanations are discussed in the main paper.(1)
8. The relations between the horizontal elutriators and cyclones were linear within the limits of dust concentration and loading examined.

Dust clouds in mines above this range are very seldom found in breathing zones of workers. Exceptions might be found close to dust sources, particularly in dry operations as in potash mining.

Acknowledgments

The authors wish to thank R. Sutarno, CANMET, Energy, Mines and Resources Canada, Ottawa, for helpful discussion leading to the development of this statistical approach.

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1 November 1984; Revised 6 February 1986

Dear Sir:

In the April 1987 issue of the Journal [Vol 48(4) 344-353 (1987)], Knight and Moore discuss the statistical analysis techniques to be used for the comparison of dust samplers.

I would like to offer two fundamental comments on this study.

When both measurements are subject to error with the following assumptions, generalizing the assumptions used in Least Squares regression analysis:

1) the average value of the errors of both measurements is zero;
2) the errors are independent;
3) the errors on Y have common variance var(ε);
4) the errors on X have common variance var(δ);
5) the errors are normally distributed.

and if we suppose that $k^2 = \mathrm{var}(\varepsilon)/\mathrm{var}(\delta)$ is known, we may use the Maximum Likelihood method to estimate the parameters of a functional relationship.[1]

If there is a linear relationship, the slope A is given by the equation in Figure 1, below, or with the notations of Equation (12) of Knight and Moore

$$A = m + (m^2 + k^2)^{1/2}$$

$$\text{where } m = \frac{1}{2A_1} - \frac{k}{2A_2}$$

$$\left(\text{and not } m = \frac{1}{2A} - \frac{k^2}{2A_2}\right)$$

$$\text{and } k^2 = \frac{\mathrm{var}(\varepsilon)}{\mathrm{var}(\delta)}$$

the ratio of the variances of the errors of measurement (and not the ratio of the variances of the measurements Y and X).

At first sight, the problem of estimating k^2 by replicated samples seems to be similar to the estimation of the constant C used by the authors to combine the arithmetic and geometric portions of the errors in weight of samples, and my feeling is that they gave up a little bit too early.

If this prior information about the relative accuracy of measurement of the two instruments is not available, other approaches to the problem of fitting a straight line have been proposed.[2]

$$A = \frac{(\mathrm{var}(Y) - k^2\,\mathrm{var}(X)) + [(\mathrm{var}(Y) - k^2\,\mathrm{var}(X))^2 + 4k^2\,\mathrm{cov}^2(X,Y)]^{1/2}}{2\,\mathrm{cov}(X,Y)}$$

Figure 1 — Slope A (linear relationship).

My second point concerns the assessment of historical dust measurements.

The problem is to estimate the expected value of the measurement Y, given by a new sampler, for every value X provided by the old instrument. That X can be subject to error doesn't matter, the known data are only the observed quantities X and not the "true" values.

This is simply a regression problem of Y upon X, needing to recreate the old operating techniques and carry out direct comparisons.

The functional relationship only helps to understand the physical relation between the pair of samplers.

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Bernard Préat
IREA
Hasselt, Belgium

Dear Sir:

I wish to thank Dr. Préat for his helpful comments on the statistical procedures used in our paper and acknowledge that these were not entirely rigorous. But I believe that they are simple procedures usable in preference to the simple linear regressions usually used, with little consideration of the errors introduced and the statistical significance of the intercepts and slopes and on some occasions non-linearity.

As to his second point on the use of the simple regression technique for assessment of historical measurements, there are two problems generally, firstly the range of measurement and secondly the weighting to be applied to each pair of observations.

In most studies the comparison measurements are conducted in "modern" lower dust levels and the results extrapolated to dust levels which were often much higher in the past than during the comparison. In many comparisons one of the pair of instruments is operating below it's useful range either because of sensitivity or because of interference by dusts not related to specific health hazard dust such as pollutants from internal combustion engines. My thesis is that these factors need to be considered and that this procedure is relatively simple and produces the best estimate available from the data. In essence the procedure "weights" the data. Both the normal linear regression and this modified approach give results which do not differ significantly in most of the measured range but give differences which can increase rapidly as one approaches the few highest measurements and proceed into extrapolation.

I believe that the use of such statistical procedures and physical understanding would reduce the wide spread of intercepts and slopes found in sampler comparisons carried out for interpretation of historical measurements in epidemiological studies such as (1,2). In the Quebec asbestos study it is apparent to one of us (GK) that in some areas such as warehouses the main difference expressed in the ratios is the difference in air pollution from internal combustion or electric powered forklifts as included by the older measurement technique.

The use of such flawed regressions for extrapolation to higher past dust levels seems most undesirable. Especially if changes in the handling techniques are not considered.

To sum up it seems that development of a physical relationship and consideration of the dust properties and possible changes is likely to avoid some misleading interpretations of the available data which could give rise to serious errors in the associated studies.

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Geoffrey Knight
Member, AIHA

Ann. occup. Hyg. Vol. 22, p. 88.
Pergamon Press Ltd. 1979. Printed in Great Britain

LETTER TO THE EDITOR

CORRELATION BETWEEN DIFFERENT MEASURES OF ASBESTOS

Sir,

The approach by DU TOIT and GILFILLAN (1977) is interesting in minimizing the effect of variance but does not discuss the significance of the resulting regression equations, which are nonlinear in terms of the original measurements and therefore represent an unlikely complex physical model, and would be of no use in understanding measurements lying outside the observed range.

We have tried similar regression techniques in comparing dust samplers (COCHRANE *et al.*, 1971) and have found them inadequate in that they do not disprove the hypothesis that a linear relationship exists between the two instruments. Our approach involved taking the regression equations log Y upon log X and log X upon log Y and obtaining the slope as a mean from the two equations following DAVIES (1961). We then found that the relationship between all the instruments was linear except those with the konimeter presumably because there was a marked subjective effect in assessing konimeter spots of different particle densities.

The other problem in comparison of field tests is the differing relative response of the instruments to dust such as air pollution, not related to asbestos and ore handling, which may cause the departure of the comparisons from passing through zero and give the 'best' hypothesis as

$$Y = a + bX$$

where Y and X are the two instruments and a is the mean value of the difference in response to pollution.

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Ann. occup. Hyg. Vol. 22, p. 89.
Pergamon Press Ltd. 1979. Printed in Great Britain

AUTHORS' REPLY

Sir,

In reply to Mr Knight's letter to you, we should like to make the following statements:

We accept that it is difficult to give clear physical interpretations to the regression equations that appear in our paper. However, extrapolation outside the observed range of measurement is always dangerous and must be undertaken with care. Therefore, in the absence of better approaches, the model generated by the observed error structures still seems the best approach at present.

We fail to understand how 'statistical techniques' can be considered 'inadequate' if 'they do not disprove the hypothesis that a linear relationship exists between the two instruments'.

The approach of Davies is old and has been superseded by methods taking account of specific error ratios. Davies' method is a special case assuming a particular error structure. As better methods are available for general error structure, we have used them.

The presence of air pollution could presumably be a factor in causing a non-zero intercept in a regression fit.

Our results were submitted in the belief that they might be of some use to convert asbestos fibre concentrations recorded by means of the konimeter and the TP to those expected with the membrane filter method. Time alone will tell whether such conversions could contribute to exposure-response studies in the asbestos field.

We are thankful for the interest shown by Mr Knight in our paper.

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R. S. J. du Toit
T. C. Gilfillan

Section 1—Chapter 10

Comparison of Respirable Dust Samplers for Use in Hard Rock Mines

by

G. Knight and E. Moore

American Industrial Hygiene Association Journal
48(4) pp354-363 (1987)

COMPARAISON D'ÉCHANTILLONNEURS DE POUSSIÈRE INHALABLE DESTINÉS AUX MINES DE ROCHE DURE

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On a comparé cinq classificateurs de poussière inhalable et onze porte-filtres, certains avec deux débits différents. Ces classificateurs comprenaient des élutriomètres horizontaux, des impacteurs, des cyclones métalliques et des cyclones en nylon. Les comparaisons ont été effectuées à la fois au laboratoire et dans des mines. On a évalué la concentration de poussière inhalable sur les filtres par pesée et, dans les cas pertinents, on a évalué la concentration de quartz par diffraction des rayons X, directement sur le filtre. Les résultats obtenus montrent que la relation entre la plupart des paires d'échantillonneurs peut être représentée par une droite passant par l'origine et que les échantillonneurs individuels recueillant le plus de poussière (l'impacteur CAMPEDS et le cyclone B.C.I.R.A.) donnent une plus grande reproductibilité pour les faibles concentrations de poussière rencontrées dans un grand nombre de mines de roche dure. Les échantillonneurs équipés d'un cyclone en nylon convenaient moins pour les mines de roche dure où la concentration de quartz est importante, mais ils étaient satisfaisants dans les mines de charbon où les normes relatives à la poussière sont plus élevées.

Am. Ind. Hyg. Assoc. J. 48(4):354-363 (1987)

Comparison of Respirable Dust Samplers for Use in Hard Rock Mines

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Five respirable dust size selectors and eleven filter holders were compared, some at two flow rates. These included horizontal elutriators, impactors, metal cyclones and nylon cyclones. The comparisons were carried out both in the laboratory and in mines. The respirable dust on the filters was assessed by weighing, and where relevant, by X-ray diffraction for quartz with the use of a direct-on-filter method. The results obtained showed that the relation between most pairs of samplers could be represented by a straight line through the origin and that the personal samplers collecting more dust — the CAMPEDS impactor and the B.C.I.R.A. cyclone — gave higher reproducibility in the low dust concentrations found in many hard rock mines. The samplers based on the nylon cyclone were less suitable for hard rock mines containing a substantial proportion of quartz, though satisfactory in coal mines with their higher dust control standards.

Introduction

Size selective sampling was shown to give the best single parameter — mass of respirable dust — in correlation with advance of pneumoconiosis as measured by lung X-ray pattern on the International Labour Organization (ILO) scale in coal mines.(1) Personal and fixed station size selective sampling has been used in many industries since.

In some of the dusty trades — hard rock mining in particular — the free silica component of the airborne dust often is considered to be significant, and the requirement is for a sampling and assessment method that will determine the weight both of the total respirable dust and its free silica component. Logically and for determination of worker exposure, personal sampling often is preferred.(2) The free silica is usually present as quartz and quantitative X-ray diffraction (XRD) assessment often is used.

The Elliot Lake Laboratory designed and tested a sampling and assessment system using gravimetry and a direct-on-filter X-Ray Diffraction (XRD) method for quartz that requires substantially less technician effort than the transfer technique recommended by the National Institute for Occupational Safety and Health (NIOSH).(3,4) Interlaboratory crosschecks have shown reasonable agreement in quartz analysis between the two methods.(5)

The Elliot Lake Laboratory first adopted the British Cast Iron Research Association (BCIRA) cyclone sampler with a 25-mm diameter silver membrane filter because it gave the highest XRD intensity to mass ratio when the various commercial size selective gravimetric samplers then available were compared in a given dust cloud.(3)

In North America, however, the 10-mm nylon cyclone has been used widely. Ontario recently has proposed a sampling and analytical system using the 10-mm nylon cyclone with a 25-mm plastic push-fit filter holder and a direct-on-filter XRD assessment of quartz and cristobalite.(6)

This report describes laboratory and field comparisons of various sampling trains and discusses a number of factors relevant to their behavior, use and assessment. Sampling pumps are not discussed, as most of them now include the essential smooth flow. In our experience, electronically controlled constant flow pumps are preferable for horizontal elutriators in the field. In most conditions, however, cyclone and impactor size selective samplers collect the same mass of "respirable" dust independent of airflow, and close attention to airflow is not required. The airflow as determined by size selection calibration rather than the mean airflow for each test should be used in calculation of the respirable dust concentration, however.

Dust Samplers Examined

In the laboratory tests at Elliot Lake, a large horizontal elutriator is used as a reference respirable dust sampler. This is the commercial unit built by Casella(7,8) modified by removal of the internal airflow control orifice, or a similar unit built by our laboratory to the British Medical Research Council (BMRC) respirable dust specification.(9) It sometimes is operated at a lower flow rate to give the best match to the American Conference of Governmental Industrial Hygienists (ACGIH) specification in simulated logarithmic normal dust clouds.(10) In all laboratory tests, the ratio of the concentrations indicated at the two flow rates has been close to the expected ratio 0.81.(11,12) All the instruments used in the laboratory and field tests are described in Table 1. Numbers 3 and 4 are the National Coal Board Mining Research Establishment (NCB/MRE) 113A gravimetric samplers manufactured by Casella. The three channel version has been modified to collect dust on a 25-mm diameter filter suitable for direct-on-filter quartz analysis. Numbers 5 and 6 are small impactor sampling heads, CAMPEDS, developed in our laboratory.(3) Numbers 7 and 8 are the BCIRA cyclone as manufactured by Casella in the U.K. or by H&H Custom in Canada.(8,13,14) In these, the precision of machining is essential; the early units varied over a range of 30% in mass of dust collected. The critical dimensions sometimes in error were entry slit width, a step at the junction between entry slit and the cylindrical bore, the length of the cylindri-

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sampler. The instrument used to connect to the Hexhlet has the value used enclosed in brackets.

- Column 7 gives the relative respirable dust concentration measured by each sampler. Again, the values in brackets in Table IV are used as connectors.
- Column 4 corresponds to Column 5 (see Tables III and V), and Column 5 is given as a relative estimate of X-ray intensity after correction for secular errors.

It is apparent from the results that the CAMPEDS and the BCIRA personal dust samplers collect substantially more respirable dust than the samplers based on the nylon cyclone.

The merit of using an electronically flow controlled pump for the NCB MRE sampler is shown by the greater reproducibility obtained in field tests 2 and 4 as compared to test 1. Test 6 is not strictly comparable because the results available gave the mean of two tests in each case.

The reproducibility observed for each instrument as an arithmetic mean of the values observed in each test is shown in Table VI and listed separately for weight of dust and for quartz.

The results suggest that in most of the varied conditions encountered in the laboratory and field tests, the CAMPEDS and the BCIRA are substantially more reproducible than the samplers based on the nylon cyclone-size selector. Even the best reproducibility observed for the nylon cyclone, 1.045, is worse than the mean CAMPEDS and BCIRA values.

Results from the CAMPEDS and the BCIRA also indicate about twice as great an XRD intensity as do nylon cyclone samplers with 25-mm diameter filters in the same dust clouds.

TABLE II
Laboratory Tests — Assessment by Weight

Code	Dust Sampler	No. of Pairs	Slope[A]	Std. Error of Estimate Geometric	Weight[B] Relative to Hexhlet	Concentration[C] Relative to Hexhlet
	Elliot Lake Laboratory Reference Hexhlet weight:					
4	*NCB MRE*	56	0.369	1.039	0.0369	0.983
	CAMPEDS Impactor					
5	Light and medium	130	0.387	1.043	0.0387	0.968
5	Heavy	28	0.0354	1.039	0.0354	0.885
6	ACGIH flow rate	27	0.0404	1.062	0.0404	0.808
	BCIRA Cyclone					
7	Serial #3	57	0.385	1.040	0.0385	1.015
	Serial #5	57	0.0421	1.032	0.0421	1.213
8	ACGIH flow rate	23	0.0418	1.049	0.0418	0.872
	Nylon Cyclone					
9	25 mm metal FH	54	0.0243	1.049	0.0243	0.714
10	25 mm plastic FH with sleeve	111	0.0210	1.101	0.0210	0.618
11	25 mm plastic FH with nipple	26	0.0193	1.050	0.0193	0.568
12	37 mm plastic FH with sleeve	24	0.0206	1.080	0.0206	0.606
13	37 mm plastic FH with nipple	49	0.0195	1.083	0.0195	0.574
16	CMDS Type I	27	0.0200	1.086	0.0200	0.500
18	CMDS Type II	26	0.0201	1.120	0.0201	0.502
	Duplicates					
1	Hexhlet	10		1.02		
5	CAMPEDS	153		1.062		
	MSHA Laboratory reference mean of no. 3, 14 and 15:					
	Limestone					
3	NCB MRE	(46)[D]	1.324	1.065	(0.05)[E]	(1.0)[E]
14	CMDS	45	0.877	1.076	0.0331	0.827
15	CMDS (1.2)	46	0.795	1.079	0.0300	1.25
	Coal					
3	NCB MRE	(36)[D]	1.415	1.070	(0.05)[E]	(1.0)[E]
14	CMDS	20	0.821	1.047	0.0290	0.725
15	CMDS (1.2)	60	0.755	1.059	0.0267	1.115
	Limestone and coal replicates					
14	CMDS	24	-	1.068	-	-
15	CMDS (1.2)	99	-	1.058	-	-

[A] Slope is ratio collected by sampler to that by reference sampler.
[B] Gives the relative amount of dust collected by a dust sampler.
[C] The relative respirable dust concentration in air given by each instrument.
[D] All the values given here refer to the expected values; however, only the mean of duplicate readings was available.
[E] The values given here assume that the NCB-MRE as used agreed with the Elliot Lake Laboratory reference sampler.

the estimated value by more than a few times the standard error, Equation 4.

The justification and evidence for this procedure are given in the companion paper.(17)

Laboratory Experiments

In the Elliot Lake Laboratory, experiments were carried out comparing dust samplers in both coal and pure quartz dust clouds in a 2.5 × 1.8 × 1.8 m walk-in test chamber. The dry coal dust is ground in a rotating plate pulverizer to pass 125 μm and fed with a Safety in Mines Research Establishment (SMRE) feeder and a compressed air ejector into the High Efficiency Particulate (HEPA) filtered airstream entering the chamber. The pure quartz cloud is prepared by feeding Ottawa silica sand to a Helme laboratory jet mill and feeding ground quartz into the HEPA filtered air stream entering the chamber.(18) The coal dust cloud is about 50% respirable (by the Hexhlet) and the quartz dust cloud 65%.

About 20 dust samplers are mounted in the chamber for each test. The airflows are controlled precisely with the use of critical orifices with inlet vacuum gauges and adjusting valves.(18) The weight of dust collected by the Hexhlet sampler (Number 1 in Table I) with its high flow rate of 50 L/min and correspondingly high precision, is used as the reference dust sampler for both total respirable dust and quartz dust. Full details of the technique are given in previous reports.(4,18)

The tests in the Mine Safety and Health Administration (MSHA) laboratory were carried out with coal mine dust samplers with their complete sampling trains in a test chamber. Both limestone and coal dust clouds were used.(19) The mean weight of dust collected by all the samplers in each test was used as that from the reference sampler in this analysis.

Field Experiments

A number of experiments have been carried out in mines, and full data for these experiments have been obtained by, or given to, the author.

Many experiments in which replicate measurements were made with one sampler were carried out to examine dust production.(20) These are referred to because the results demonstrated that the differences between replicates had both arithmetic and geometric error terms. The differences in very light samples gave a normal distribution of errors on a linear basis, whereas a logarithmic transformation was required to produce a normal distribution for the heavy samples. The arithmetic portion of the errors, about 0.1 mg, is associated mainly with slight damage or contamination occurring in loading and unloading filter holders and partly with weighing errors. The geometric portion, about 10%, is associated with sampling and differences in mean dust cloud concentration to which nominally side-by-side dust samplers are exposed in the variable dust clouds. The measured variability of the replicates was represented most simply by the following expression: 95% of the pairs of side-by-side samples had weight differences between them of less than

$$0.1 + 0.1\ W \text{ mg} \qquad (5)$$

where W is the mean weight of the pair of samples, and

$$0.01 + 0.1\ Q \text{ mg} \qquad (6)$$

where Q is the mean weight of quartz assessed by XRD on the pair of samples. These led to estimates of 1 mg and 0.1 mg, respectively, for the constant C in Equation 3.(17)

Test 1 was carried out in two uranium mines by an experienced mine technician with the Elliot Lake Laboratory Equipment. The NCB MRE dust samplers were ten years old and the pump performance was below the standard achieved when new; their sample weights were not included in the reference. This test also produced a substantial number of duplicate samples, the analysis of which is included.

Test 2 was carried out in the same uranium mines by a team of experienced technicians. Two preliminary tests carried out in the laboratory test chamber in a pure quartz dust cloud were included. Test 3 was a comparison between the existing and the proposed dust sampler carried out by Provincial Mines Inspectors in gold mines. Test 4 was carried out as a part of a major study in iron ore surface mines and mills in Labrador.(21) Test 5 also was carried out in the iron ore surface mines and mills. It consisted of a total of 450 pairs of samples. Test 6 was carried out in coal mines.

Results

The statistical analysis of the comparisons between dust samplers is presented in detail in a second paper.(17) Summaries are presented here in Tables II to V, where the number of pairs, the slope relative to the reference sampler and the geometric standard error from the transformed regression Equation 4 are given. For each test where the Hexhlet was not the reference sampler, the instrument used as an intermediate reference has its values given in brackets. The relative concentrations for measurement of quartz are not given in Tables III and V because appropriate calibration would give the same values as in the last column of Tables II and IV.

The significance of each column in Tables II and IV is as follows:

- Column 4 — slope by weight is the ratio of the weights of dust collected by each sampler to the reference sampler as determine by the statistical analysis.
- Column 5 is the standard error of the estimate for each sampler. Unfortunately the error in the reference sampler is still necessarily included, but as this is constant in each set of experiments, the samplers still can be compared within each set. The differences between sets contain terms that can be attributed to reference instruments, conditions and experimenters.
- Column 6 enables comparison of weights collected by samplers when the Hexhlet is not used as the reference

TABLE I
Descriptions of Dust Samplers

Code	Sampler	Resp. Dust Spec.	Use	Flow Rate L/min	Size Selector	Connection	Filter Holder	Filter Diam. mm	Filter Type	Pump Lab.	Pump Field
1	Hexhlet	BMRC	Lab Reference	50.0	Elutriator	Integral	Metal screw	55	GF A	CO'	-
2	Hexhlet	ACGIH	Lab Reference	33.6	Elutriator	Integral	Metal screw	55	GF A	CO	-
3	NCB-MRE	BMRC	Coal Mine	2.5	Elutriator	Integral	Metal clamp	55	GF A	SDP''	SDP
4	NCB-MRE	BMRC	Mod. for quartz	1.875	Elutriator	Integral	Metal clamp	25	Ag	CO	EFC'''
5	CAMPEDS	BMRC	Hard rock mine	2.0	Impactor	Integral	Metal spring	25	Ag	CO	SDP
5A	CAMPEDS	BMRC	Hard rock mine	2.0	Impactor	Integral	Metal spring	25	Ag	-	EFC
6	CAMPEDS	ACGIH	-	2.5	Impactor	Integral	Metal spring	25	Ag	CO	-
7	BCIRA	BMRC	Foundries and Hard rock mines	1.9	Cyclone	Integral	Metal screw	25	Ag	CO	SDP
7A	BCIRA	BMRC			Cyclone	Integral	Metal screw	25	Ag	CO	EFC
8	BCIRA	ACGIH		2.4	Cyclone	Integral	Metal screw	25	Ag	CO	-
9	10 mm Nylon	ACGIH	Can. Grain Studies	1.7	Plastic cyclone	Sleeve	Metal clamp	25	Ag	CO	SDP
10	10 mm Nylon	ACGIH	Ont. Hard Rock Mine	1.7	Plastic cyclone	Sleeve	Plastic pressfit	25	Ag	CO	SDP
10A	10 mm Nylon	ACGIH	Ont. Hard Rock Mine	1.7	Plastic cyclone	Sleeve	Plastic pressfit	25	Ag	-	EFC
11	10 mm Nylong	ACGIH	Ont. Utilities	1.7	Plastic cyclone	Nipple	Plastic pressfit	25	Ag	CO	-
12	10 mm Nylon	ACGIH	General N. Amer.	1.7	Plastic cyclone	Sleeve	Plastic pressfit	37	Ag	CO	SDP
12A	10 mm Nylon	ACGIH	General N. Amer.	1.7	Plastic cyclone	Sleeve	Plastic pressfit	37	Ag	-	EFC
13	10 mm Nylon	ACGIH	General N. Amer.	1.7	Plastic cyclone	Nipple	Plastic pressfit	37	Ag	CO	-
14	Coal Mine Dust (Type I)	MSHA	U.S. Coal Mines	2.0	Plastic cyclone	Sleeve	Plastic pressfit	37	Plastic	CO	ADP
15	Coal Mine Dust (Type I)	BMRC	-	1.2	Plastic cyclone	Sleeve	Plastic pressfit	37	Plastic	CO	SDP
16	Coal Mine Dust (Type I)	MSHA	-	2.0	Plastic cyclone	Sleeve	Plastic pressfit	37	Ag	CO	-
17	Coal Mine Dust (Type II)	MSHA	U.S. Coal Mines	2.0	Plastic cyclone	Nipple	Plastic pressfit	37	Plastic	CO	-
18	Coal Mine Dust (Type II)	MSHA	-	2.0	Plastic cyclone	Nipple	Plastic pressfit	37	Ag	CO	-

'CO - critical orifice, smooth;
''SDP - smoothed diaphragm pump;
'''EFC - electronically flow controlled

cal portion of the bore, and an off center vortex finder. Manufacture requires special tool and jig fabrication followed by careful inspection to maintain size selection performance within a range of 5%. Our stock includes units showing some differences, and the duplicate samplers are analyzed separately.

Numbers 9 to 17 are various versions of sampling heads based on the nylon cyclone. Number 9 was a modification of the MSA sampler for direct-on-filter quartz analysis used in the Canadian grain industry study.(15) Numbers 10 and 11 are the heads proposed by Ontario Ministry of Labour for dust sampling in industries with silica.(6) These differ in the connection between the nylon cyclone and the filter. Numbers 12 and 13 are common variants in North America. Numbers 14 and 17 are two types of commercial coal mine dust samplers approved by NIOSH.(16) Number 15 is Number 14 run at a lower airflow. Numbers 16 and 18 are these two units with the cassettes reloaded with silver membrane filters for direct-on-filter analysis.

The Statistical Analysis Technique

The statistical analysis technique is treated in a second paper.(17) It was shown that the data do not meet the assumptions on which the least squares method of statistical linear regression is based and that transformations and corrections should be made. It was found that a straight line through the origin and the point defined by the means of the transformed dependent and the independent variables was a good model for the relationship between most pairs of dust samplers examined.

$$y = Ax \qquad (1)$$

where y is the weight of dust (or quartz) collected by the sampler under test;
x is that collected by the reference sampler; and
A is the slope.

$$A = e^{[\Sigma \ln(y + C) - \Sigma \ln(x + C/A)]/N} \qquad (2)$$

where C is a constant and N the number of pairs.

The exception was comparisons with the CAMPEDS (Number 5) when heavy samples, above 4 mg, were included and in quartz analysis of sampler Number 11.

A transformation was found essential to produce common variance in the errors and produce consistent estimates of error for comparison of dust samplers. The transformation used is:

$$Y = \ln(y + C);\ X = \ln(x + C/A) \qquad (3)$$

where Y is the dependent variable;
X is the independent variable; and
C is a constant used to unify the arithmetic and geometric portions of the error.

The standard error of the estimate, SE, of Y is found with the use of departure of the values from the estimated values in Equations 2 and 3 as:

$$SE = e^{[(\Sigma Y^2 + \Sigma X^2 + N \ln^2 A - 2 \ln A\, \Sigma Y + 2 \ln A\, \Sigma X - 2 \Sigma XY)/(N-2)]^{0.5}} \qquad (4)$$

where Σ implies summation over the N values of Y or X; and
N-2 is the appropriate number of degrees of freedom.

Elimination of data points as outliers was based on evidence of sampling errors or physical damage and departure from

TABLE III
Laboratory Tests — Quartz Assessed by Direct-on-Filter XRD

Code	Dust Sampler	No. of Pairs	Std. Error of Estimate Geometric	Relative[A] XRD Intensity
4	NCB MRE	20	1.147	0.0300
	CAMPEDS Impactor			
5	3MRC	52	1.081	(0.0387)
6	ACGIH	20	1.101	0.0450
	BCIRA Cyclone			
7	Serial No. 3	18	1.081	0.0383
	Serial No. 5	20	1.099	0.0413
	Nylon Cyclones			
9	25-mm metal FH	20	1.176	0.0202
10	15-mm plastic FH with sleeve	40	1.210	0.0218
11	25-mm plastic FH with nipple	18	1.141	0.0265
12	37-mm plastic FH with sleeve	19	1.108	0.0117
16	CMDS type I	20	1.138	0.0098
18	CMDS type II	19	1.154	0.0075

[A] Gives the relative X-ray intensity from samples collected by each sampler

Comparisons of all the results for weight suggest that the Elliot Lake Laboratory results and field tests 1 to 5 show good agreement between the impactor and cyclone size selective samplers. The results for the horizontal elutriator are more variable. The respirable dust assessment given by the horizontal elutriator is some 15 times more sensitive to airflow error than cyclones or impactors, as determined by comparison of samplers with and without 10% flow rate errors. Thus, the variable performance probably was caused by small systematic errors in airflow calibration in each test

TABLE IV
Field Tests — Assessment by Weight

Code	Dust Sampler	No. of Pairs	Slope[A]	Std. Error of Estimate Geometric	Weight[B] Relative to Hexhlet	Concentration[C] Relative to Hexhlet
Test 1 — Reference means of no. 5 and 7						
3	NCB MRE	67	1.576	1.173	0.0571	1.18
5	CAMPEDS	75	1.070	1.028	(0.0387)	(0.968)
7	BCIRA	76	0.926	1.026	0.0359	0.945
Test 1 — Included replicates						
3	NCB MRE	23	-	1.101	-	-
5	CAMPEDS	30	-	1.044	-	-
7	BCIRA	31	-	1.034	-	-
Test 2 — Reference mean of no. 4, 5 and 7						
4	NCB MRE	31	0.903	1.024	0.0340	0.900
5	CAMPEDS	61	1.027	1.032	(0.0387)	(0.968)
7A	BCIRA	33	1.017	1.016	0.0383	1.008
10	Nylon 25 mm	16	0.601	1.045	0.0226	0.665
12	Nylon 37 mm	17	0.621	1.048	0.0234	0.688
Test 3 — Reference no. 7A BCIRA					(0.0383)	(1.008)
12A	Nylon 37 mm	29	0.600	1.236	0.0230	0.676
Test 4 — Reference mean of no. 4, 5A, 7A and 12A						
4	NCB MRE	64	0.988	1.059	0.0337	0.898
5A	CAMPEDS	64	1.135	1.042	(0.0387)	(0.968)
7A	BCIRA	68	1.224	1.059	0.0418	1.100
12A	Nylon 37 mm	67	0.680	1.101	0.0232	0.819
Test 5 — Reference sampler no. 7A (BCIRA)						
5A	CAMPEDS	414	0.915	1.068	(0.0387)	(0.968)
Test 6 — Reference mean of no. 3, 14 and 15						
3	NCB MRE	57	1.529	1.059	(0.0500)	(1.00)
14	CMDS	55	0.724	1.045	0.0237	0.592
15	CMDS (12 L/m)	57	0.748	1.045	0.0245	1.020

[A] Slope is ratio collected by sampler to that by reference sampler.
[B] Gives the relative amount of dust collected by a dust sampler.
[C] The relative respirable dust concentration in air given by each instrument.

TABLE V
Field Tests — Quartz Assessment by Direct-on-Filter XRD

Code	Dust Sampler	No. of Pairs	Std. Error of Estimate Geometric	Relative[a] XRD Intensity
Test 1 — Reference means of No. 5 and 7				
5	CAMPEDS	70	1.061	(0.0387)
7	BCIRA	72	1.065	0.0298
Test 1 — Included replicates				
5	CAMPEDS	30	1.071	-
7	BCIRA	28	1.084	-
Test 2 — Reference means of No. 4, 5 and 7				
4	NCB MRE	31	1.060	0.0304
5	CAMPEDS	61	1.066	(0.0387)
7	BCIRA	35	1.068	0.0381
10	Nylon 25	17	1.155	0.0163
12	Nylon 37	17	1.086	0.0102
Test 4 — Reference mean of No. 4, 5A, 7A and 12A				
4	NCB MRE	64	1.074	0.0298
5A	CAMPEDS	62	1.057	(0.0387)
7A	BCIRA	63	1.062	0.0372
12A	Nylon 37	66	1.087	0.0096
Test 5 — References BCIRA No. 7A				
5A	CAMPEDS	416	1.098	(0.0387)

[a] Gives the relative X-ray intensity from samples collected by each sampler.

because of factors such as surface barometric pressure, average depth in the mine and, for the studies by the other laboratory (Table II) and Field Test 6, problems arising from the pump oscillations not being completely smoothed and affecting the flowmeter reading in the original Casella units used.(28) Airflow calibration tests on one of the Elliot Lake Laboratory's old 113As demonstrate that the Rotameter shows errors due to incompletely damped oscillations and also that the calibrations of the three 113A Rotameters tested were not correct at normal temperature and pressure.

Discussion

The statistical analysis of the comparisons between dust samplers shows the errors associated with most of the samplers examined are reasonable at less than 15% as long as the sampler would collect more than 1 mg of dust at the given target level and for an appropriate sampling time. When the amount of dust to be collected is less than this, the proportional error rises, and the advantage of the instruments collecting more dust becomes apparent in their greater reproducibility.

TABLE VI
Summary of Dust Measurement Reproducibility

	Standard Error of Estimate	
	for Weight	for Quartz
NCB MRE	1.070	1.087
CAMPEDS	1.039	1.076
BCIRA	1.043	1.080
Nylon cyclone samplers	1.079	1.139

For quartz assessment with its target level of about 0.1 mg, the advantages of the CAMPEDS or BCIRA are apparent for the direct-on-filter method. While it is possible that the transfer technique developed in the United States could reduce the arithmetic portion of the error, there is no published evidence to support it.

In hard-rock mines, the choice of size selection specification should be considered. The International Standards Organization (ISO) recommends that either the BMRC or the ACGIH specification can be chosen with matching choice of the standard.(6,22,24) For direct-on-filter XRD assessment of quartz, the important factor is the weight of quartz dust per unit area, as long as the filter is as large or larger than the X-ray beam. The BMRC specification then has the following advantages as compared to the ACGIH

1) airflow lower;
2) dust bed coarser and more open; and
3) resistance to airflow substantially less.

In other words, when the pump performance is the limiting factor, a substantially thicker bed can be produced with a substantial increase in XRD response. This is particularly useful for the direct-on-filter XRD assessment of quartz. The transfer to second filter technique can be used to concentrate the dust from a larger diameter filter in sampler and obtain a greater XRD intensity. We chose the direct-on-filter technique, however, because it requires less technician time and skill as the transfer step is eliminated.

Other important factors in dust sampling in hard rock mines are:

1) dust deposition pattern on the filter;
2) filter holder sealing;
3) coarse dust penetration to second stage filter;
4) reproducibility of sampling train;
5) materials of construction;
6) linearity in the relationship between dust samplers - weight; and
7) linearity in the relationship between dust samplers - quartz.

Dust Deposition Pattern

In the direct-on-filter XRD assessment of quartz, the beam examines only a portion of the filter; thus, the assessment depends on the dust deposition pattern. Obviously, while a uniform deposition would be desirable, no samplers achieve it. Tables II and III show that differences of 40% in the ratio of XRD response to mass of quartz exist for the samplers with 25-mm diameter filters.

The quartz assessment of an uneven deposit can be calibrated by collection of samples in a pure quartz dust cloud. If the pattern varies from sample to sample, however, the assessment is subject to random errors.

No variations in dust pattern from one type of sampler have been observed in samples collected in the laboratory, but large variations have been seen in batches of samples submitted by some mines. The manifestations are in variations in the presence and intensity of thicker (darker) center or rings near the edge visible on the dust deposits. The most

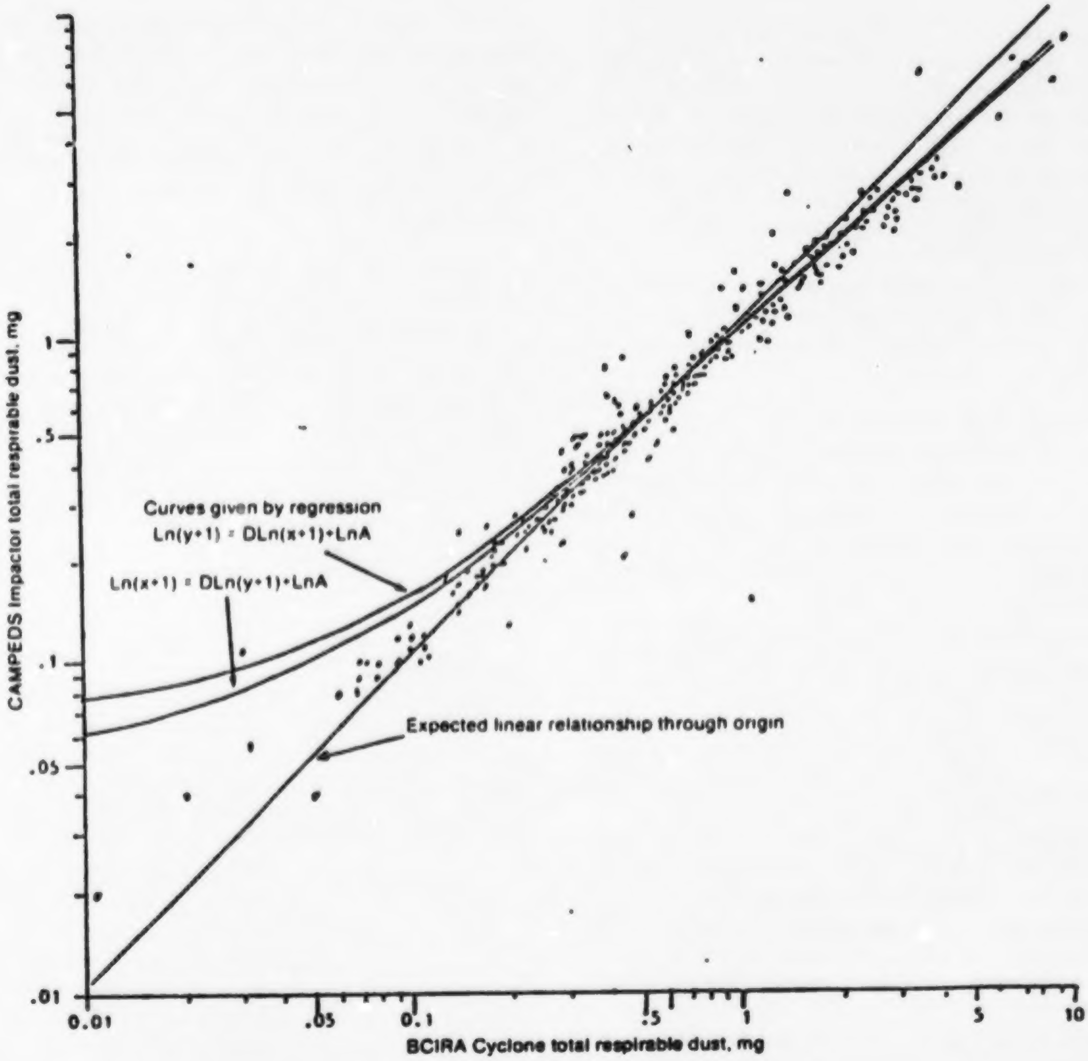

Figure 1 — Non-linearity in one dust sampler.

marked variations have been in batches of samples collected with nylon cylones.

It is recommended that a small diameter tube should not be used to attach the filter holder to the nylon cyclone vortex finder, as it apparently is prone to produce variations in dust deposition pattern and as the central peak would lead to extra difficulties in correction for absorption on heavy samples.

Filter Holder Sealing

Leakage of air around the edge of filters in pressfit filter holders has been reported.(24) On many filters received for quartz analysis, it has been noticed that dust deposits extend into the supposedly clamped area, presumably because of edge leakage. This has occurred on up to one third of the samples shipped by one mine using pressfit filter holders. Notifying the technician reduced the problem to about one filter per batch. A change in technician led to a recurrence of the high rate, however.

Leakage of dust laden air around the edge of the filter leads to three problems: 1) low indicated respirable dust concentration; 2) an even lower indicated quartz dust concentration, because the dust on the edge while weighed is not included in the XRD assessment; and 3) penetration of dust into the pump with resulting increased maintenance.

The press-fit filter holders commonly are used with the nylon cyclones; the leakage has not been found in samples collected in our laboratory when threaded clamps are used on the pressfit holders. The BCIRA screw-up filter holders only occasionally demonstrate edge leakage, and the CAMPEDS spring seal has never shown this fault.

Coarse Dust Penetration

Rough handling of dust samplers can lead to the transfer of collected coarse dust from the first stage to the second stage filter. When the transfer occurs after, or near the end, of sampling, loose flocs of dust can be seen on the surface of the smooth dust deposit on the second stage filter. One often can remove these flocs without damaging the dust deposit by holding the filter surface vertically with tweezers and gently tapping the tweezers. This problem was found fairly frequently on personal samples collected with the BCIRA cyclone and with the nylon cyclone. Laboratory experiments in which the BCIRA cyclone was turned upside down over a weighing dish after the filter was removed and then tapped gently have transferred on average about 20% of the weight of the coarse dust collected.

The problem has not been seen with the CAMPEDS impactor, where both a labyrinth seal and adhesive in the coarse dust collection area were introduced to reduce the possibility of dust transfer.

Reproducibility of Sampling Train

To achieve adequate reproducibility in size selection, cyclones require greater precision in machining than is normal machine shop practice.(25,26) Adequate precision can be achieved by special tooling and jigs or by moulding. Inspection is probably necessary to reject size selectors with flawed critical surfaces.

Horizontal elutriators require careful attention to detail to bring about the even airflow among the channels that is required for achieving the expected size selection.

The CAMPEDS impactor has not shown any variation between sampling heads, probably because the main critical dimensions are two orifices and the drills used are sufficiently precise, as purchased.

The authors have found that a number of technicians have overtightened the off-center clamp on one commercial nylon cyclone sampler, causing the entry orifice to open up and resulting in a gross overestimate of the respirable dust. In the authors' view, any size selector is acceptable only if its component parts are attached rigidly to each other by screws, clamps or glue.

The filter holder can affect the dust depositing on the second filter (see, for instance, the difference between samplers 9 and 10), and the complete sampling train needs verifying for size selection performance.

Materials

Charge build-up in the size selector has been suggested as a cause of variable size selection performance.(27) Various experiments with the nylon cyclone and a copy in metal have come up with conflicting results.(27-31) The authors believe that some of the variability in the results achieved in sampler comparisons in the authors' laboratory chamber can be attributed to electrostatic phenomena associated with the great differences in relative humidity occurring between summer and winter in the northern laboratory. It would appear prudent to build cyclones and filter holders from metal or semi-conducters rather than insulators to minimize such effects.

Linearity: Weight

The linearity of the relation pairs of samplers by weight was examined specifically in one part of this study where a factorial experiment was carried out at two levels of three variables: medium and high dust concentration; medium and high dust loadings; and coal and quartz dust clouds. The only significant nonlinearity detected was with the CAMPEDS. The nonlinearity was correlated most closely with the amount of coarse dust collected and occurred when the cone of dust building up under the orifice approached the plane of the orifice. Presumably, this condition increases the dust impaction.

A power function obtained with the use of regression on the transformed variable, (Equation 3), gives a better fit for the heavier samples as shown for the Labrador field study, Test 5 in Figure 1. The level at which departure from linearity occurred was reached only in 5% of the fixed station comparisons and in about 2% of all the samples taken at the iron ore plants. It has been reached less frequently in full shift personal sampling in underground hard rock mines. The CAMPEDS size selector, however, can be modified to increase the coarse dust holding capacity. Modified units are, at the time of writing, awaiting test.

Linearity: Quartz

The linearity found for most dust samplers and the nonlinearity found for heavy CAMPEDS samples, related to collection of dust and presumably would be reflected in the weight of quartz collected and presented to the X-ray beam. This section is not concerned with that, but only with the additional effects specifically related to the quartz assessment.

A nonlinearity has been noticed before and attributed to entry of dust into the pores of the 5 μm pore size silver membrane, where it is hidden from the X-ray beam.[illegible] It was shown that this was not associated with significant penetration through the filter, and it was presumed that when the dust bed built up, the extra quartz was presented to the XRD beam. This nonlinearity has been presumed to be related to the total dust on the filter rather than just the quartz fraction. The effect is about halved for 3 μm pore size silver membranes and not found for 1.2 or 0.8 μm pore size. All quartz results in this report have been corrected appropriately for this effect based on the calibration curve originally obtained with the BCIRA cyclone. The distribution was shown to be far from normal without a transformation or with a logarithmic transformation in the companion report.(17) In Figure 2 it can be seen that the differences between the measured quartz and the estimate when Equations 1 and 2 are used for CAMPEDS, Number 5, is distributed almost normally. The slight dip at 5-10 mg on the scale (2-4 mg on the filter) for the CAMPEDS would not be noticeable if the high set at 25 mg and the low set at about 8 mg were removed. Most of the other samplers show similar distributions. The nylon cyclones with nipples, Numbers 11 and 13, however, show a marked dip; the dip for Number 11 is shown in Figure 2. This dip is probably an artifact caused by the very high central peak found with this sampler and the use of a nonspecific calibration for the light sample correction factor and by the spread in the central peak as the dust layer builds up.

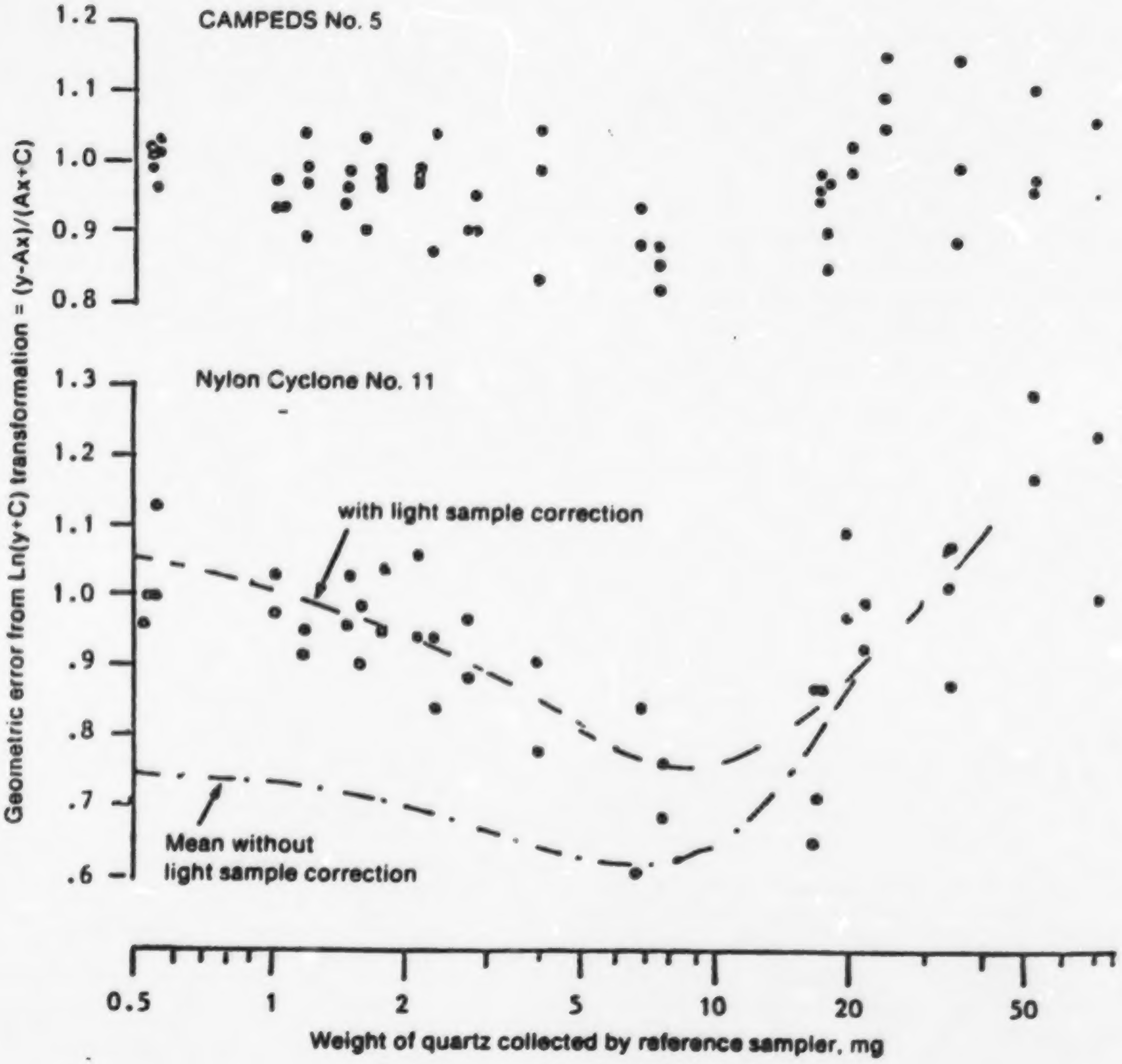

Figure 2 — Non-linearity of quartz estimates.

Conclusions

1) Statistical analyses of comparisons between samplers seldom meet the assumptions on which the least squares method is based and corrections should be made. Any departures found from the expected relationship — usually linear through the origin — should be justified by significance tests or physical basis (from the companion paper Reference 17).
2) The total dust sampling system, including gravimetric and quartz assessment, requires calibration. This study shows that the size selection calibration can be carried out satisfactorily by comparison with a reference sampler even with the use of side-by-side samplers in the field, and that direct-on-filter quartz determination introduces nonlinearity because of dust bed effects, which has been allowed for with the samplers normally used.
3) The 20% difference in indicated respirable dust concentrations found between different sampling trains using the cylon cyclone, even though probably largely due to the filter holder and its connection, illustrates the problem attributable to the lack of a defined reference sampling train to the ACGIH specification. The BMRC specification has adequate reference dust samplers available in commercially available or self-constructed horizontal elutriators.
4) The BCIRA cyclone or CAMPEDS impactors with gravimetric and quartz assessment collect more dust more reproducibly in the same dust cloud and thus are more suitable than nylon cyclones for measuring the small quantities found in hard rock mines.
5) The CAMPEDS impactor underestimates respirable dust when the collected coarse dust exceeds about 4 mg. The dust holding capability should be improved in the next production run.
6) The CAMPEDS impactor is the most suitable dust sampler of those examined for personal sampling in hard rock mines, especially when its coarse dust hold-

ing capacity has been increased. Unfortunately, because of insufficient demand it is no longer available commercially.

7) The nylon cyclone is adequate for full shift sampling with gravimetric assessment in mines with target levels similar to the coal mines, 2 mg/m^3.
8) Size selectors are acceptable only if the component parts are screwed, clamped or glued solidly together. The off center adjustable clamp over size selector and filter used in one commercial unit is not adequate.
9) Push fit filter holders are not satisfactory in that they require either a press or a high standard of technician training to avoid significant edge leakage.

Acknowledgments

The authors wish to thank R. Kowalchuk, J. Hoar, R. McClellan, R. McCabe, A Frattini, H.N. Treaftis and others for the collection of and permission to use the data from the field and other laboratories in this analysis.

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1 November 1984; Revised 8 September 1986

Letters to the Editor

Dear Sir:

The paper "A Filter Cassette Assembly Method for Preventing Bypass Leakage"[1] highlights a recurrent problem we have in receiving samples on silver membranes from mines for quartz analysis by x-ray diffraction.

The indication of bypass leakage, dust on supposedly clean edge of filter, occurs quite often. The two last batches of filters for instance had 70% and 30% respectively of edge deposits.

Whilst we point out the problem to the appropriate technician and the leakage rate is reduced to an acceptably small proportion, it often returns when the technician is changed, which is often as frequently as yearly in many mines.

This problem appears to be associated mainly with the press fit plastic filter holders in the 25 and 37 mm diameter sizes, popular in North America. It would appear to be a better solution to redesign the filter holder to achieve a more technician proof assembly. We have found that assemblies using elastomeric O rings and spring pressure are leakproof.

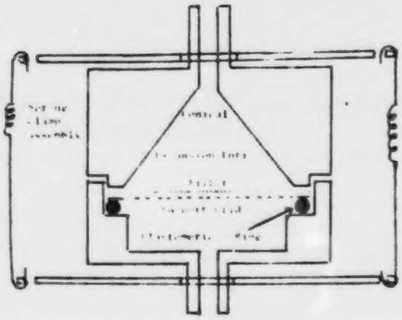

Figure 1 — The essential elements of an improved filter holder.

The proportion of samples, with damage due to the filter holder, has been very low in many thousands of samples collected by a number of technicians in mines. Our sampling head has been described elsewhere.[2] The essential details of the filter assembly are shown in Figure 1. In addition a shrink fit band or tape can be used to keep dirt out.

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Ann. occup. Hyg., Vol. 00, No. 0, pp. 000–000, 1984
Printed in Great Britain

0003-4878/84 $3.00+0.00
Pergamon Press Ltd
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LETTER TO THE EDITOR

CORRELATION BETWEEN MEASUREMENTS BY MRE Casella 113A) AND CYCLONE DUST SAMPLERS

Sir,

Dr Verma in his paper 'Inhalable, Total and Respirable Dust: A Field Study' (*Ann. occup. Hyg.*, **28**, 163–172, 1984) found poor correlation between measurements by the MRE and by the 10 mm mylon cyclone samplers, and suggests that size distribution, environmental effects and orientation are the cause. This seems unlikely in view of the good correlations found between the nylon cyclone and total or inhalable dust measurements.

The effect agrees with the poor correlation we found in our last field study using the then 10-yr-old 113As with their own pumps. We have found much better correlations with other samplers in later field trials when we used electronically flow-controlled pumps and thus we attribute the poor correlation to inadequate airflow control. The respirable mass collected by horizontal elutriators samplers is some 10 to 20 times more sensitive to airflow errors than that by cyclone samplers.

In airflow calibration attempts on the 113A we have found that the rotameters read about 0.10 l min^{-1}. low, corrected to normal temperature and pressure on a smooth flow; and that they read high when installed in the 113A, presumably because of some remaining airflow pulsation after the flow damper. I wonder if any reader can elucidate these problems.

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